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FEDERAL RADIOLOGICAL MONITORING AND ASSESSMENT CENTER

FRMAC MONITORING AND ANALYSIS MANUAL

SAMPLE PREPARATION AND ANALYSIS

VOLUME 2

U.S. DEPARTMENT OF ENERGY
NEVADA OPERATIONS OFFICE
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Bechtel Nevada
REMOTE SENSING LABORATORY

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PREFACE

Under the Federal Radiological Emergency Response Plan (FRERP), the federal agencies with statutory responsibilities for dealing with radioactive materials emergencies, have agreed to coordinate their emergency response efforts. In the event of a potential or existing major radiological incident, the Department of Energy/Nevada Operations (DOE/NV) has been charged with establishing and operating the Federal Radiological Monitoring and Assessment Center (FRMAC). The FRMAC is to provide coordinated federal assistance in the offsite areas to the impacted state(s) and the lead federal agency (LFA) responsible for regulation and/or operation of the accident site.

The Monitoring Division (MD) of the FRMAC is responsible for a significant portion of the assistance provided. These responsibilities include: field radiation monitoring, environmental sampling, and radioanalysis. The MD coordinates the efforts of all federal, state, and local or tribal agency staffs working with the FRMAC to provide scientifically defensible data of acceptable quality.

This manual has been prepared by the United States Environmental Protection Agency (EPA) Office of Radiation and Indoor Air (ORIA), Radiation Sciences Laboratory-Las Vegas for the U.S. Department of Energy under Interagency Agreement Number DE-A108-91-NV10963.

The DOE/NV has responsibility for maintaining the master of all FRMAC manuals. Please provide comments on this manual to: FRMAC Program Manager, Emergency Operations Office, P.O. Box 98518, Las Vegas, Nevada 89193-8518.

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INTRODUCTION

When the Federal Radiation Monitoring and Assessment Center (FRMAC) responds to a radiological accident, monitoring, sampling, and radioanalysis support will arrive from a number of different sources. The respondents providing this support will, in all likelihood, have received varying levels of training and will have experience with a variety of monitoring, sampling, and radioanalysis equipment and procedures. The Monitoring Division (MD) requires that the methods used during the FRMAC response be uniform and consistent to ensure that the derived data are reliable and defensible. It is important that an acceptable and established set of standard operating procedures (SOPs) be followed by all respondents for any monitoring, sampling, and radioanalysis activity conducted under the direction of MD. This manual provides SOPs for sample processing and radioanalysis and will be used by MD during a FRMAC response to a radiological accident.

Early in an emergency, monitoring data will be urgently needed as a basis for protective actions. FRMAC emergency response procedures are intended for use in processing relatively large numbers of samples in the shortest possible time. Therefore, in some instances, they represent a compromise between precise analytical determinations and determinations for emergency response activities.

These methods are applicable to the FRMAC mobile laboratory. They are not mandatory for all mobile laboratories which may be associated with a specific FRMAC deployment. The methodologies employed by any participating laboratory are acceptable, if they are documented, meet FRMAC requirements specific to that deployment, and produce analytical data of acceptable quality.

In the early stages of an emergency, when the impact on the health and safety of the public is not well defined, the resources dedicated to quality assurance (QA) activities will be sufficient to assure acceptable quality of the data. As the emergency stabilizes, QA activities will increase correspondingly.

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SURVEY OF FRMAC LABORATORY CAPABILITIES

This procedure outlines the steps to be taken by the FRMAC Laboratory Supervisor to determine what analytical capabilities are available from the on-site mobile laboratories and their personnel. The information will be critical for both the Assessment Division (AD) and Monitoring Division (MD) Managers in order to set meaningful priorities for the collection and analysis of environmental samples. The Laboratory Supervisor will use this information to efficiently program the sample preparation and analytical operations for optimal throughput with adequate detection limits.

Scope and Application

The evaluation of field capabilities will include:

- Inventory of the instrumentation of each laboratory:

  types, sizes, and number of detectors; precalibrated geometries; counting efficiencies; background counting rates; computer data reduction capabilities

- Determination of ability to adjust to changing conditions and analytical needs; e.g., recalibrating for new geometries, introducing additional nuclides into spectrometry libraries

- Demonstration of adequate personnel to perform and validate the required analyses

Responsibilities

Individual laboratory managers are responsible for providing information to the MD Manager on their instrumentation, capabilities, and personnel resources. The Quality Assurance (QA) Officer will assist in the transfer of information, setting of priorities, and documentation of the ongoing performance of the laboratories. The operator of each mobile laboratory will be given a copy of this procedure by the QA Officer and will be asked to complete and return the form within a specified period.

Instrument and Capability Summary

The form to be completed by laboratories can be found in Appendix D.

PREPARATION OF QUALITY ASSURANCE SAMPLES FOR FRMAC FIELD LABORATORIES

	Marinelli Beaker		Plastic Bottle 500 mL	Charcoal Cartridge	Air Filter		Widemouth Jar 400 g
	1.0L	3.5L			2"	4"	
Water	X	X	X				
Milk	X	X	X				
Air				X	X	X	
Soil							400 g
Vegetation	100 g	1.0 kg					

Nuclides:
Alpha: Th-230, Pu-238, Pu-239, Am-241
Beta: H-3, Sr-90, Cs-137, I-131
Gamma (60-1836 keV): Am-241, Cd-109, Co-57, Ce-109, Hg-203, Sn-113, Sr-85, Cs-137, Co-60, Y-88.
Also Cs-134, I-131.

Scope and Application

This methodology is used under field conditions to prepare QA samples to assess the capability of mobile laboratories to perform:

- gamma spectrometric analyses on a variety of samples
- gross alpha and gross beta analyses on air filters collected during an emergency response situation

This describes the preparation of samples which are too bulky or contain nuclides that are too short-lived to be kept on hand in a repository that would be shipped to the emergency site on short notice.

- Examples: water samples to be gamma counted in various geometries (outlined above) or air filters, charcoal cartridges, or other samples listed above that may contain I-131.

QA samples allow for the initial assessment and documentation of continuing performance of these instruments used in the field:

- gas proportional alpha/beta counters

- liquid scintillation counters
- sodium iodide or high-resolution germanium detectors for gamma-ray spectrometry

Summary of Method

Availability

Instrument calibration standards will be available to all participants at the FRMAC. All required radionuclides are available commercially in NIST-certified standards.

Preparation

An ampule containing radionuclides is opened, while working in a fume hood, and the solution transferred to a secondary glass container. Dilutions are made from the stock nuclide which will be used to prepare QA samples containing lower levels of activity.

- **Water samples** are prepared by diluting the stock nuclide or the diluted stock nuclide to volume using calibrated automatic pipets.
- **Air filter samples** are prepared by transferring a measured volume of radionuclide to the filter medium and air drying in a desiccator or under a heat lamp.

Supplies

Absorbent paper
Absorbent wipes
Adjustable presision pipettes, 20 mL, 100 mL, 1 mL, 5 mL, with tips
Ampules, 5 mL polyethylene
Beakers
Chemical carriers
Chlorine bleach
Desiccators
Deionized water
File (triangular or flat)
Filters: glass fiber, paper, Teflon, (2", 5", 8"x 10")
Gloves
Heat lamps
Hydrochloric acid (1 M)
Iodine cartridges
Lab coat
Labels, radioactive and regular (unmarked)
Lap-top computer with spreadsheet capability
Liquid scintillation vials, 20 mL with poly-seal caps
Marinelli beakers 1.0 L, 3.5 L
Miscellaneous containers for counting: aluminum cans, jars, cubitainers, etc.
Planchets, planchet holders 2", 4"
Radionuclides in 5 mL ampules
Safety glasses (goggles)
Sissors
Sealable plastic bags

Sodium carbonate
Sodium iodide
Stoppered vials
Swipes
Transfer pipets
Transfer vials
Volumetric flasks
Waste container, lined with plastic bag with proper label

Procedure

Records

Supply a copy of the NIST-traceable certification file.

Refer to Appendix A, Example 1, for an example of a NIST file. As an alternative, you may use the table in Example 2 as a guide.

Transfer and Dilution of Stock Nuclides

Take proper laboratory precautions before working with these radioactive standards:

- Wear a suitable lab coat, gloves, safety glasses, and TLD badge.
- Before any of the radioactive standards are opened, designate a waste container for the empty ampules.

1. Check neck of 5 mL ampules for colored band; if band is present, ampules can be opened without scoring ampule.
   - Place ampule in small plastic bag and gently snap off top at neck.
   - Aspirate solution into transfer vial (plastic baby bottle) and add to volumetric flask containing matrix solution. Use gravimetric correction to account for amount of primary standard used.
2. Before proceeding, refer to special precaution section for I-131, if I-131 solutions are to be used.
3. Prepare dilutions of stock radionuclides for preparation of lower activity QA samples.
   - Prepare diluted solution to same HCl molarity as original NIST solution.
4. For each solution prepared from the stock solution, record spike volume, dilution volume, sample geometry, and certificate file reference date (see Appendix A, Examples 2 and 3).

Commercial NIST-traceable mixed gamma samples generally include a combination of Am-241, Cd-109, Co-57, Cs-137, and Co-60, Hg-203, Sn-113, Y-88, which provide gamma rays at several energies ranging between 60 and 1332 keV.

Gamma-ray geometry standards should be prepared so that at least 10,000 counts are accumulated in each gamma peak of interest in the allotted calibration collection time (a minimum of 10 minutes).

Preparation of samples containing I-131

- Solutions must be alkaline (pH 10 or greater)
- Evaporated samples (air filters, charcoal cartridges) must contain the iodine in the iodate chemical form $[IO_4-]$.

1 Working in a fume hood, open ampule containing approximately 2 mCi of I-131 and aspirate solution into transfer vial.

2 Add the solution to a 100 mL volumetric flask containing: 50 mL water, 1 mL chlorine bleach, 1 g sodium iodide, and 1 g of sodium carbonate.

3 Dilute to the mark with water, stopper, and agitate.

4 Prepare dilution from the 100 mL I-131 stock solution using a solution containing 1 mL bleach and 1 g each of sodium iodide and sodium carbonate per 100 mL.

5 Prepare QA air filters or charcoal cartridges by pipetting I-131 stock solution or diluted solution directly on the filter or cartridge and allowing the sample to dry in a desiccator.

Filters containing I-131 with other nuclides can be prepared if the nuclide solutions containing acid are allowed to air dry thoroughly on the filter media before adding the solution containing the I-131.

Note

Gross alpha and gross beta air filters are prepared using Th-230 and Cs-137. Multiply the Cs-137 activity by 1.10 to compensate for conversion electrons produced in the decay of its short-lived progeny, Ba-137m.

Water samples containing tritium are not acidified.

Calculations

Radionuclide Decay Corrections

The activity of individual stock nuclides at the time of QA sample preparation is calculated from the following equation:

$$A(t) = A(r) \times \{EXP[-0.693 \times (t-r)/T(1/2)]\}$$

where

$A(r)$ = Activity of stock nuclide on reference date, r, in nCi/g

$t-r$ = Difference in days between the date of preparation, t, and the reference date, r, of the nuclide

$T[1/2]$ = Half-life of the nuclide in days

Precision and Accuracy

The radionuclides used for the preparation of the FRMAC QA samples are purchased from commercial suppliers. All nuclides are accountable through a NIST-traceable program.

Uncertainties in pipetting, dilution, and reproducing geometrical arrangements are not expected to exceed 10%. Verify activity against precalibrated detector.

INTRODUCTION OF QUALITY ASSURANCE SAMPLES TO MOBILE LABORATORIES AT A FRMAC EXERCISE OR EMERGENCY RESPONSE

This procedure describes the routine use of QA samples during FRMAC responses or exercises. The QA Officer is responsible for preparing and introducing samples of known activity to mobile laboratories as a means of demonstrating and documenting laboratory capability.

Scope and Application

The actual performance characteristics of the individual laboratories must be demonstrated after the analytical needs of the FRMAC have been defined as a function of the scenario or emergency situation and the potential capabilities of the mobile laboratories on site have been established from the capability survey, "Survey of Field Laboratory Capabilities." This performance must be demonstrated as soon as possible after establishment of the FRMAC.

The analysis of QA samples with known composition will allow for evaluating laboratory performance and the subsequent validation of FRMAC data. The analysis of QA samples will also provide early warning for laboratories having problems and allow for corrections which will lead to comparability of data among all analytical operations.

Responsibilities

The Monitoring Division (MD) QA Officer is responsible to have available, or be able to prepare on site, sufficient QA material to demonstrate the validity of the analytical operations. See "Preparation of Quality Assurance Samples for FRMAC Field Laboratories." The QA Officer will work with individual laboratory managers and personnel to ensure that all of the instrumentation and appropriate technologies are used. The QA Officer will consult with the MD Manager and the Laboratory Supervisor to periodically introduce QA samples into routine analytical operations.

Procedure

1 Laboratories check radiation counting instruments as soon as the FRMAC has been established and the QA samples are ready for distribution.

At least two concentration levels will be examined to demonstrate proper instrument calibration.

2 Using gamma spectrometers, analyze QA samples containing several nuclides with energies that approximate nuclides that could be represented in an accidental release or incident.

3 MD Manager makes initial determination of instrument performance after consulting with the Laboratory Supervisor and the QA Officer.

- Determines if individual instruments need to be recalibrated or if correction factors need to be applied for consistency between laboratories or between instruments.

4 QA samples will be introduced into the sample flow at a rate decided by the MD Manager, the AD Manager, and the Senior Science Advisor. These spiked samples will be introduced at the rate of approximately one per 20 samples analyzed.

5 Background counts will be performed routinely. The frequency and counting time will be determined by the number of samples to be analyzed, the variation of the background level, and the possibility of contamination.

Reference

U.S. Environmental Protection Agency. Data Quality Objectives for remedial response actions, EPA/540/G87/003. March 1987.

SAMPLE RECEIVING PROCEDURES

Scope and Application

This method describes the setup and operation of the FRMAC sample receiving line (SRL), including the monitoring and receiving of samples from the field. All samples associated with the FRMAC operation will be received through the SRL.

Summary of Method

SRL personnel set up a Sample Receiving Area where samples are received from field monitoring personnel, packaged, and surveyed. Priorities and paperwork are checked and samples placed in a holding area.

Each sample received is considered contaminated and handled accordingly.

- Sample is placed into a clean outer plastic bag.
- Survey reading is taken and recorded on the bag.
- Any additional labels indicating priorities for further sample processing and analysis are affixed to the outer bag.
- Security Seal is checked to be sure it is intact.
- Sample is placed into a holding area, segregated by activity, priority, media, collection location, or any other parameter.
- **All paperwork is properly checked before the field monitoring team is released.**

Personnel at the Sample Control Area are notified that the sample is ready for transfer.

Supplies

anti-contamination clothing (boots, hoods, gloves, coveralls)
cardboard/card stock (for area signs)
Chain-of-Custody forms/tape
chairs, folding, portable
clipboards

cubitainers, 1-gallon (or 4 Liters), with caps and plugs
garbage bags and ties
gloves, vinyl
labels, assorted, permanent adhesive
logbooks
marking pens, permanent, any surface
masking tape, 2-inch
note pads, 8.5 × 11 inches, ruled
paper towels/wipes
pens, ball-point, permanent
plastic bags, 15 × 26 × .004 inches, clear
plastic sheeting, .004-inch minimum
plastic sealable bags, clear, 4 × 4 inches
plastic sealable bags, clear, 6 × 6 inches
radwaste bags
respirators (if needed)
Sample Control Forms
sample storage boxes
scissors
survey instruments
swipes
tape- radioactive
tweezers/forceps, large
work gloves, cloth/leather
work table(s), folding, portable
yellow cloth/duct tape, 2-inch
yellow rope/tape (to mark area boundaries)

Procedure

1 Set up a Sample Receiving Area.

- Cover work tables with plastic sheeting.
- Use yellow rope/tape to clearly mark areas.
- Make directional signs from cardboard.
- Monitor entire work area periodically for contamination.
- If possible, provide seating (with a protective coating) for waiting field monitors.

Field Team Leader will not leave until all samples, and associated paperwork, have been properly checked.

2 When receiving a double-bagged sample,

- Verify with field monitor that activity written on the inner bag is correct.

- Determine whether MD and/or Laboratory Supervisor has issued any instructions for the prioritization of this sample.
- Collect and check sample paperwork for completeness.

  Paperwork (inside the outer bag) must be readable through the unopened bags.
- **Check Security Seal to make sure it is intact.**
- **Process urgent samples first.**

3 Survey sample using appropriate instrument.

4 Using two people, place double-bagged sample into a clean outer plastic bag and seal with tape.

- Be careful not to contaminate the new plastic bag or any hot/cold personnel.
- The sample collection form inside the sample bag must remain readable.

5 Mark the survey instrument reading on the bag.

6 Affix any additional required labels.

Use of additional labels is determined at the MD briefing, and may include labels designating priority, location, activity, etc.

7 Place the triple-bagged sample into a holding area, segregated by activity, priority, media, location of collection, or any other parameter decided upon in previous MD briefings.

8 Notify Sample Control that the sample is ready for transfer and log-in.

Note

Expedite urgent and high priority samples.

SAMPLE CONTROL

Scope and Application

The operation of Sample Control is described here. All samples associated with the FRMAC operation will be processed through Sample Control for screening and entry into the LIMS identification system.

Supplies

Sample Control Forms

Procedure

1 Samples are obtained from the sample holding area of the Sample Receiving Line (SRL) in the order of their arrival.

2 Priority samples are handled first, in the order of 1) "urgent" and 2) "high priority." All samples are moved to the holding area of the sample control facility.

3 Samples designated as "hot" (red sticker) may also have priority designations. These samples will be kept separate from others.

These samples will be stored away from FRMAC personnel to achieve ALARA conditions.

4 The sample readings written on the inner bags, or on the labels, will be verified.

5 Sample Control Form completion.

- Laboratory Supervisors identify required analyses and the laboratories to analyze the samples.
- The original is retained by Sample Control, entered into the LIMS, and filed for later reference.
- The pink copy remains with the sample for delivery to Sample Preparation.

6 The Laboratory Supervisors will periodically notify participating laboratories of the samples and their required analyses.

7 The samples and their pink Sample Control Forms will be picked up and delivered to Sample Preparation or directly to the laboratories. A specific courier may be assigned this task.

Note

Be careful at all times that no contamination occurs in Sample Control.

PREPARATION OF FRMAC FIELD SAMPLES FOR GAMMA RAY SPECTROMETRY

	Marinelli Beaker		Plastic Bottle 500 mL	Charcoal Cartridge	Air Filter		Widemouth Jar 400 g
	1.0L	3.5L			2"	4"	
Water	X	X	X				
Milk	X	X	X				
Air				X	X	X	
Smears				X	X	X	
Soil							400 g
Tissues							100 g
Vegetation	100 g	1.0 kg					

Procedure: Samples are prepared for transport to, and analysis or storage at, FRMAC laboratories. Many of the samples will be shipped to participating fixed laboratories for precise analytical determinations.

Scope and Application

For the preparation of liquid, air, vegetation, soil, smear, and tissue samples at any FRMAC activity, these procedures are recommended. They are appropriate for preparation for screening and analysis of samples at FRMAC mobile laboratories.

The Laboratory Supervisor is responsible for utilizing the combined capabilities of mobile and fixed laboratories to provide analytical data of known and sufficient quality to meet FRMAC data quality objectives (DQOs) at each phase of the emergency.

Supplies

Sample preparation facilities should be stocked with at least the following supplies:

Analytical balance
Bench Top Absorbent Material
Blank adhesive labels
Can sealer
Chain-of-Custody forms
Concentrated nitric acid

Disposable gloves, goggles, other protective laboratory clothing
First Aid Kit
Fixed Facility forms
Forceps, scissors, knives
Formaldehyde
Heavy plastic bags, tape
Note pads, 8.5 × 11 inches, ruled
Marking pens
Monitoring instruments
Paper towels/wipes
Pruners
Radwaste containers
Refrigeration facilities
Sample Control Forms
Sample containers (such as Marinelli beakers, teflon lined aluminum cans, or containers of other appropriate calibrated geometries)
Scissors
Smears
Sodium Hydroxide
Tape–radioactive

The facility also must have adequate ventilation to protect personnel from airborne particulates that may pose a hazard during operations.

Procedure

FRMAC mobile laboratories are responsible for specification or estimation of data quality (i.e., precision, accuracy, etc.) for all data that they generate and for achieving FRMAC DQOs within their stated capabilities. Transportation of collected environmental samples to obtain precise analytical data will be decided upon by the AD and MD Managers as the exercise progresses. Sample preservation information will be provided with each sample type listed below.

All samples obtained from Sample Control must have:

- *Sample Control Form.*
- Intact *Security Seal* on the outer bag.

If these items are not in order, immediately notify the Laboratory Supervisor, set the sample aside, and await further instruction.

Aliquots are referenced to the original sample number.

Liquid Samples

All bags containing liquid samples are visually inspected before being accepted into the sample preparation facility. If leakage has occurred, necessary precautions are implemented to prevent contamination of the facility, other samples, and personnel.

If a sample cannot be prepared without danger of contamination, notify the Laboratory Supervisor and set the sample aside.

Milk

Milk samples designated for shipping or archiving should be refrigerated. The addition of formaldehyde will interfere with future wet chemistry analyses, especially radiostrontium.

If radioiodine analysis is needed on these samples, they should be gamma-ray counted immediately at the mobile laboratory or transported without delay to a fixed laboratory due to the short half-life of I-131 (8.04 days). The samples are shipped in their original cubitainer (decontaminated and swiped) if no visible damage has occurred. If the seal has to be broken, proper chain-of-custody procedures must be followed. A plug must be inserted into the cubitainer for transportation.

Water

Water samples designated for shipping or archiving are preserved by the addition of concentrated nitric acid (10 mL/4 L sample) to ensure all dissolved substances remain in solution.

Note

If either tritium or I-131 analysis is required, aliquots must be removed before the sample is acidified.

Caution

As with all caustic chemicals, extreme caution must be exercised in the use of acid. The samples are shipped in their original cubitainers if no visible damage has occurred. However, proper chain-of-custody procedures must be followed since the seal will have been broken to preserve the samples. The cubitainers must be plugged, decontaminated, and swiped before transportation. Proper documentation concerning acidification must also be included on all appropriate labels and forms.

Preparation for Gamma-Ray Analysis

1. Measure samples into standard geometry 1.0 L (kg) or 3.5 L (kg) Marinelli beakers, or another geometry that has been gamma-ray efficiency calibrated with a water matrix calibration standard. The containers must have predetermined tare weights or volume fill lines.

2. Distribute sample volumetrically or by weight measurement. If sufficient sample does not exist to fill the counting container, use

an aliquot and dilute with distilled water to the fill line of the container. Aliquot volume (or weight) must be recorded so that the concentration can be calculated. Generally, liquid samples will not be smaller than 100 mL.

3 The deposition activity per unit area is important for snow samples. Carefully measure the mass or volume of the entire sample and record with the mass or volume of the aliquot so that the surface activity can be back-calculated. Include the original surface area in the calculations.

4 Clearly mark sample control number on containers Fasten lids securely to prevent contamination of the laboratory and sample loss.

Reuse containers only if they can be cleaned to remove contamination after sample analysis. Otherwise, discard them. All containers that have been cleaned must be inspected by gamma-ray analysis, by the direction of the Laboratory Supervisor, to confirm the absence of radioactive contamination.

Vegetation Samples

Caution

Submission of vegetation samples to the sample preparation facility requires great care to protect the facility and personnel from possible contamination. Due to the non-uniformity of vegetation samples, sticks, branches, and other sharp objects may puncture the plastic bags during sample transportation. This will increase the chances of contamination through tears in the bags. Also, vegetation samples are often dusty and pose an additional hazard that laboratory workers must consider.

Samples that are to be shipped or archived will generally require no preparation. However, if the Laboratory Supervisor determines that spoilage may be a problem, as with fruits or vegetables, the samples are frozen or refrigerated. The samples are shipped in their original containers if no visible damage has occurred, although proper chain-of-custody procedures must be followed if the seals are broken.

Note

If an immediate or emergency gamma-ray screen is required, refer to "Preparation of Vegetation for Gamma-Ray Analysis" below.

All vegetation samples collected by the field monitoring teams are likely to contain loose particle deposition. Therefore,

- Do not clean, shake, or wash samples before preparation for counting.

- Remove vegetation from the plastic bag and carefully pack it into a standard 1.0 L or 3.5 L Marinelli beaker, or other suitable container that has been calibrated with a water matrix or suitable vegetation gamma-ray efficiency calibration standard.

Leafy Vegetation

Shred the sample if the leafy vegetation is not flexible enough to easily be pressed into a gamma-ray analysis container. Shred by hand, using scissors, or with a knife. Be careful not to remove any of the particle deposition.

Do not blend samples.

Other Vegetation

Other vegetable matter such as roots, fruits, vegetables, etc., may be collected to measure the extent of deposition of activity or the later absorption of activity. The consistency may vary, so processing the material to a form suitable for gamma-ray analysis may require cutting the portions into manageable pieces.

- For screening of deposited activities, do not wash the matter before adding it to the counting containers.
- For determining absorption, washing and/or peeling is required.

  Do not wash and/or peel unless the original screening indicates that activity is above the prescribed minimum detectable activity. Refer to "Gamma Emitting Radionuclides in FRMAC Samples."

Preparation of Vegetation for Gamma-Ray Analysis

If an emergency gamma-ray screen is needed on a field vegetation sample, use the following procedure if the sample is not too large.

1. Make sure that the
   - Outer plastic bag is secure and has been properly decontaminated and surveyed.
   - Detector and the inside of the shield are properly protected in case the plastic liners are torn in this procedure.
2. Carefully place the vegetation sample into the detector shield around the detector, making sure the shield can be closed.
3. Count the sample for 10 minutes, or long enough to detect any gamma contamination.

- If detectable activity is present, go to the Step 4, below.
- If no detectable activity is present, report results and archive sample.

4 Pack the vegetation into a standard 1.0 L or 3.5 L Marinelli beaker, or other suitable container that has been calibrated with a water matrix or suitable vegetation gamma-ray efficiency calibration standard. The container must have had its tare weight predetermined before use.

5 Once the container is filled to near capacity, determine and record the weight of the material.

6 Tape the lid securely on the container to prevent contamination of the laboratory.

Soil Samples

Soil samples are of use primarily to determine the surface deposition activity of the area from which they are collected.

1 Thoroughly mix the sample.

2 Weigh and record the sample.

3 Determine the container's tare weight before use.

4 Clearly mark sample control number on container.

Air Filter Samples

Air filter samples are inspected for physical damage within the delivery envelope. If the filters are damaged, the Laboratory Supervisor is informed.

1 Remove the filters from the envelope with tweezers or forceps and place in a labeled counting planchet (alpha/beta counter), or in a labeled plastic bag (gamma detector).

Be careful that no collected surface material is lost from the filter during the transfer.

2 Clean the tweezers or forceps following the transfer of each filter.

8" × 10" Filters Most radioanalytical laboratories are equipped to handle only 2" and/or 4" air filter samples. However, if the laboratory has the ability to analyze high volume 8" × 10" filters,

- Fold in quarters before placing in the counting holder.

- Be careful not to lose collected material on the surface of the filter that is disturbed during folding.

3 When a charcoal cartridge canister is received from the field, take it out of the delivery envelope and inspect it.

4 Before gamma-ray analysis, protect the detector from contamination. Place the canister onto the detector resting on a suitable sample holder, or place it in a plastic bag before it is put onto the detector.

5 Count the charcoal cartridge in both the up and down positions for the same length of time.

6 Average and report the resulting activities, including counting uncertainties or the MDA values.

Animal Tissue Samples

Animal tissue samples present both radiological and biological hazards to all personnel handling them.

Note

If an animal tissue sample must be counted at a mobile FRMAC facility, the following procedure can be used providing the sample is not too large. Make sure the outer plastic bag is secure and has been properly decontaminated and surveyed.

1 Ensure the detector and the inside of the shield are properly protected in case the plastic liners are torn in this procedure.

2 Carefully place the animal tissue sample into the detector shield around the detector, making sure the shield can be closed.

3 Count the sample for 10 minutes, or long enough to detect any gamma contamination.

- If detectable activity is present, go to the Step 4, below.
- If no detectable activity is present, add preservative, report results, and archive sample.

4 Physically prepare all tissue sample for sealing in aluminum tins. Depending upon the consistency of the samples, this may require cutting the tissue into pieces.

- Record the total mass of the sample weighed into the tin.
- Put in enough sample that the tin is nearly filled (if possible).
- Add water to bring the weight or volume to the full capacity of the tin.

5 Refrigerate or freeze samples at the FRMAC. Seal and clearly mark cans with their sample control numbers for shipping, archiving, or gamma-ray analysis at a fixed radioanalytical laboratory.

The gamma-ray analysis of the animal tissues will probably provide only a rough estimate of the actual activity and should only be used in decisions which can tolerate the associated uncertainty. A suitable calibration standard using an aluminum can geometry for this matrix must be performed prior to gamma-ray tissue analysis. It is highly recommended that these animal tissue samples be sent to fixed radioanalytical laboratories for precise analytical measurements, including ashing, if necessary.

ANALYSIS OF GAMMA EMITTING RADIONUCLIDES IN FRMAC SAMPLES

Analyte:	Gamma Emitting Radionuclides
Procedure:	The sample is placed in a standard geometry and counted with a Germanium (Ge) or Sodium Iodide NaI(T*l*) detector for gamma rays. Gamma rays are resolved by gamma spectrometry and the concentrations of specific radionuclides are calculated.
Matrix:	Water, Milk, Air, Soil, Vegetation, Smears, and Tissues
Accuracy:	± 30% at MDA

Scope and Application

This method covers the non-destructive measurement of gamma ray-emitting radionuclides in a variety of environmental matrices by high purity germanium (Ge) and/or sodium iodide (NaI[T1]) detector spectrometry. It is applicable for nuclides emitting gamma or × rays with energies greater than 5–10 keV for Ge detectors, and 50 keV for NaI detectors. The upper energy range is usually established on the order of 3000 keV. However, it should be noted that the proper detector calibration ranges are a function of the nuclides that are included in the various gamma calibration standards.

The method can be used for 1) qualitative and quantitative determinations using Ge detectors or for 2) screening and semi-quantitative and semi-qualitative determinations using NaI detectors. Exact quantitation using NaI is possible for single nuclides or when the gamma emissions are limited to a few well-separated energies.

Energy and efficiency calibrations for each detector should be pre-established before FRMAC deployment, or after arrival at the FRMAC.

The following mixed gamma calibration standards, along with their appropriate calibration certificates, will be available to all participating laboratories that request their use.

Charcoal cartridge
1.0 L Marinelli beaker
2" air particulate filter
3.5 L Marinelli beaker

4" air particulate filter
500 mL plastic bottle
Soil (400 g in widemouth jar)
Vegetation (100 g in a 1.0 L Marinelli beaker)

Nuclide libraries for gamma spectrometry should contain the recommended nuclides and gamma-ray lines recommended by the Nuclear Regulatory Commission NUREG 1028. Computer software should be available to list the contents of the library and to add additional nuclides and gamma-ray lines to the library for peak search routines. An example of a gamma-ray library is presented in Table 1 of this method.

Capability to perform calibrations of new geometries in the field should be available. The MD of FRMAC will have available a number of mixed gamma emitting standards for calibration, each containing 8–10 nuclides.

Summary of Method
(EPA Resources)

The sample or a homogenized aliquot of the sample (soil, water, or vegetation) is put into a standard geometry and placed on or next to the appropriate detector and counted for a period of time sufficient to meet the sensitivity criteria established by the Laboratory Supervisor. Minimum detection concentrations are calculated for specific nuclides as required.

The spectral data are stored electronically and processed using computer-based peak search and integrating routines to determine gamma ray line intensities and specific nuclide concentrations. Concentration values are accompanied by a value estimating the uncertainty in the measurement of the specific nuclide.

Sample Handling and Preservation

Samples for gamma analysis are prepared according to "Preparation of FRMAC Field Samples for Gamma Ray Spectrometry."

Sample density and homogeneity are also factors which may affect quantitative results. Errors can be minimized by preparing efficiency calibration standards in a similar matrix and placed in identical counting positions on the detector. The charcoal cartridge sample should be counted in both the up and down positions and the results averaged.

Interferences

When counting gamma emitters with a NaI(Tl) detector, significant interference may occur. If gamma rays from different radionuclides are similar in energy, peaks will overlap and will be difficult to resolve. Higher energy gamma rays which predominate may completely mask minor, less energetic photopeaks for both Ge and NaI detectors. The use of Ge detectors minimizes resolution problems.

Cascade summing may occur when nuclides emit gamma rays in coincidence. Be aware that certain nuclides will be underestimated when using peak searching routines. Extremely high counting rates can cause spectral shifts and distortion with both counting systems. Increase the distance between the sample and the detector, or screen and dilute samples, when necessary, to avoid this problem and the potential contamination of the counting equipment and possibly laboratory personnel.

Apparatus

1 Large volume Ge (>25%) or NaI detector with optional shielding. Planar or Be end window Ge detector for low energy gamma ray measurement in air filters or smears (2" diam.).

2 Supporting electronics.

3 Data acquisition system including a multichannel analyzer with a computer which collects data, provides a visual display, stores spectral data, and provides hard copy printouts of analysis results.

The computer should have software to perform peak integration and nuclide search and activity calculation routines for high resolution Ge spectrometry or linear regression or other peak unfolding routines for low resolution NaI counting.

The analyzer should be able to provide 2048 or 4096 channels for Ge spectral data storage or 256 or 512 channels for NaI spectrometry. The computer should also be able to generate a table of MDA values for all nuclides in the computer library used with the germanium detector, or list specific nuclides of interest defined by the sample matrix counted.

Procedure

1 Count the sample and perform peak search and integration routines and the peak background subtraction routine if necessary. Calculate activity concentrations and associated counting uncertainties of the nuclides identified. Calculate MDAs for nuclides of interest not identified in the sample spectrum and identify possibilities for lines detected in the spectrum which are not listed in the gamma ray library.

2 Count non-homogeneous samples, such as radioiodine cartridges or soil samples representing a given deposition area, a

second time in an identical but inverted geometry; average the results of the two counts.

3 Note on analytical reports samples containing high suspended solids or sediments and estimate uncertainty in the measurement.

4 Retain all raw data.

5 Each mobile Laboratory Supervisor verifies results before submitting them to the MD Laboratory Analysis Specialist for review.

Calculations

Calculate individual radionuclide concentrations from:

$$\text{Conc.} = A/(T \bullet Eff \bullet Y \bullet S \bullet D \bullet CF)$$

where

A = peak area above continuum (counts)

T = count length (seconds)

Eff = fractional detector efficiency at photopeak energy (counts/gamma)

Y = fractional gamma abundance (gammas/disintegration)

S = sample size

D = decay correction (collection to count) (EXP(–0.693 dT/T(1/2))

CF = Conversion factor (1.00 for Bq, 0.037 for pCi)

The 2-sigma counting uncertainty is calculated from:

$$\text{Error}(\%) = 200 \bullet \text{SQRT}(A+2C)/A$$

where

C = photopeak area below continuum (counts)

Precision and Accuracy

Functions contributing to precision and accuracy are:

- Sensitivity achievable in the analysis

- Counting parameters, such as count time and geometry
- Sampling uncertainties.

To estimate precision and accuracy, analyze an appropriate number of spiked samples, blind samples, duplicate samples, and replicates. These analyses will be conducted at a frequency that will ensure an acceptable level of data quality.

Quality Assurance

Energy calibration should be verified during review of results of each sample.

System background spectra should be collected using an empty sample container and the same count time as used for samples. Background spectra should be collected at the beginning of each work shift and whenever contamination of counting equipment is known or suspected.

Calibrations

Perform energy calibration using standards of energies comparable to the isotopes of interest. For example:

Detector	Type of Gamma Source	keV/channel
Ge	multi-line	0.5–1.0
NaI (256-channel)	3–4 line (Am-241, Cs-137, Co-60)	5–10

Adjust the amplifier gain and the analog-to-digital converter zero offset to locate the various peaks in the appropriate channels.

1 Perform efficiency calibrations using a NIST-traceable mixed gamma source for Ge spectrometry or a series of individual calibrated nuclides for NaI counting.

   Calibrations, preferably in the same matrix to be analyzed, must be performed for each of the standard geometries before they can be used in routine counting.

2 Calculate efficiencies at several well-spaced gamma energies (photopeaks) over the normal range of analysis.

3 Count the standards for sufficient time to accumulate at least 10,000 counts in each photopeak area.

4 ► **Plot the counting efficiency vs gamma energy** for each counting geometry-detector combination. Use the curve for sample analysis.

Calculate counting efficiency from:

$$Eff = R/(A \cdot Y)$$

where

R = net count rate, counts per second (integrated counts in the photopeak above the base line continuum divided by the counting time in seconds),

A = activity of the radionuclide added to the standard geometry container (disintegrations per second [Bq]) corrected for decay to the time of count.

Y = fractional yield or abundance of the specific gamma ray for the given nuclide (gammas/disintegration).

Examples

- **Table 2** presents an efficiency data calculation sheet.
- **Efficiency curves** for a 3.5 L and a 2" filter on a 30% Ge detector are presented in Appendix B, Figures 1 & 2.

Efficiencies for non-standard geometries can be estimated by normalizing the straight line portion of the log-log efficiency vs energy curve of an established geometry-detector combination.

1 Prepare a sample containing Cs-137 and Co-60 in the new geometry and determine the counting efficiencies at 661, 1173, and 1332 keV as described above.

2 Calculate the ratios of the three efficiencies for the two geometries.

3 Adjust the straight line portion of the standard curve for slope and intercept to produce a curve for the new geometry.

Linear regression interference factors for resolving NaI spectra are determined by calculating the counting efficiencies of interfering nuclides in the region of interest of the nuclide.

References

U.S. Department of Energy, *Environmental Measurement Laboratory Procedures Manual*, HASL-300 27th Ed. (1990).

Krieger, H.L, and E.L. Whittaker, *Prescribed Procedures for the Measurement of Radioactivity in Drinking Water*, U.S. Environmental Protection Agency, EPA600/4-80-032 (1980).

1994 Annual Book of ASTM Standards Vol 11.02, Water(II), American Society for Testing and Materials, Philadelphia, Penn. (1994).

U.S. Nuclear Regulatory Commission, *NUREG Guide 1028* (1994).

U.S. Environmental Protection Agency, *Radiochemical Analytical Procedures for Analysis of Environmental Samples*, U.S. Environmental Monitoring and Support Laboratory, EMSL-LV-0539-17, Las Vegas, Nevada (1979).

Table 1. Sample Gamma Ray Library

Library Title	
Library file name	$DISK1:[USER]SHORT.NLB;1
Date printed	25-JAN-1 990 09:07:58.70
Number of nuclides	46
Number of lines	118

Nuclide Name	Half-Life	Nuclide Type	Key Line	Energy	Abundance
Be-7	53.44D	activation		477.59 keV	10.42 %
Na-22	2.60Y	activation		1274.50 keV	99.90 %
K-40	1.28E+09Y	natural		1460.81 keV	10.67 %
Cr-51	27.70D	activation		320.08 keV	9.83 %
Mn-54	312.70D	activation		834.83 keV	99.97 %
Co-57	270.90D	fission		122.06 keV 136.48 keV	85.51 % 10.60 %
Co-58	70.80D	activation		810.76 keV	99.40 %
Fe-59	44.63D	activation		1099.22 keV 1291.56 keV	56.50 % 43.20 %
Co-60	5.27Y	activation		1173.22 keV 1332.49 keV	100.00 % 100.00 %
Zn-65	244.40D	activation		1115.52 keV	50.75 %
Se-75	11 9.78D	activation		121.11 keV 136.00 keV 264.65 keV 279.53 keV 400.65 keV	16.70 % 59.20 % 59.80 % 25.20 % 11.40 %
Rb-83	86.20D	activation		520.41 keV 529.64 keV 552.65 keV	46.00 % 30.30 % 16.40 %
Sr-85	64.84D	fission		513.99 keV	99.27 %
Y-88	106.60D	fission		898.02 keV 1836.1 keV	93.40 % 99.38 %
Nb-95	35.06D	fission		765.79 keV	99.81 %
Zr-95	64.02D	activation		724.18 keV 756.72 keV	43.70 % 55.30 %

Nuclide Name	Half-Life	Nuclide Type	Key Line	Energy	Abundance
Ru-103	39.35D	fission		497.08 keV	89.00 %
Ru-106	368.20D	fission		621.84 keV	9.80 %
Cd-109	464.00D	fission		88.03 keV	3.72 %
Sn-113	11 5.100D	fission		255.12 keV	1.93 %
				391.69 keV	64.90 %
Sb-124	60.20D	fission		602.71 keV	97.87 %
				645.85 keV	7.26 %
				722.78 keV	11.10 %
				1691.02 keV	49.00 %
Sb-125	2.77Y	fission		176.33 keV	6.89 %
				427.89 keV	29.33
				463.38 keV	10.35
				600.56 keV	17.80 %
				606.64 keV	5.02 %
				635.90 keV	11.32 %
I-126	12.93D	fission		388.63 keV	29.10 %
Sb-126	12.40D	fission		666.33 keV	99.62 %
				695.00 keV	99.62 %
				697.00 keV	29.00 %
				720.50 keV	53.80 %
				856.80 keV	17.60 %
I-131	8.04D	fission		284.30 keV	6.05 %
				364.48 keV	81.20 %
				636.97 keV	7.26 %
				722.89 keV	1.80 %
Ba-133	10.50Y	fission		79.62 keV	2.55 %
				81.00 keV	33.00 %
				276.40 keV	6.90 %
				302.84 keV	17.80 %
				356.01 keV	60.00 %
				383.85 keV	8.70 %
Cs-134	2.06Y	fission		563.23 keV	8.38 %
				569.32 keV	15.43 %
				604.70 keV	97.60 %
				795.84 keV	85.40 %
				801.93 keV	8.73 %

Nuclide Name	Half-Life	Nuclide Type	Key Line	Energy	Abundance
Cs-136	13.16D	fission		176.55 keV	13.56 %
				273.65 keV	12.66 %
				340.57 keV	48.50 %
				818.50 keV	99.70 %
				1048.07 keV	79.60 %
				1235.34 keV	19.70 %
Cs-137	30.17Y	fission		661.65 keV	85.12 %
Ce-139	137.66D	fission		165.85 keV	80.35 %
Ba-140	12.79D	fission		162.64 keV	6.70 %
				304.84 keV	4.50 %
				537.32 keV	25.00 %
La-140	40.22H	fission		328.77 keV	20–50 %
				487.03 keV	45.50 %
				815.85 keV	23.50 %
				1596.49 keV	95.49 %
Ce-141	32–50D	fission		145.44 keV	48.40 %
Ce-144	284.30D	fission		133.54 keV	10.80 %
Eu-152	13.60Y	activation		121.78 keV	28.40 %
				244.69 keV	7.49 %
				344.27 keV	26.50 %
				411.11 keV	2.21 %
				443.98 keV	3.11 %
				778.89 keV	
				867.32 keV	4.16 %
				964.01 keV	14.40 %
				1085.78 keV	10.00 %
				1112.02 keV	13.30 %
				1407.95 keV	20.70 %
Hg-203	46.60D	activation		279.19 keV	77.30 %
Pb-210	22.30Y	natural		46.50 keV	4.00 %
Pb-214	26.80M	natural		74.81 keV	6.33 %
				77.11 keV	10.70 %
				241.90 keV	7.46 %
				295.21 keV	19.70 %
Ra-224	3.64D	natural		240.97 keV	3.69 %

Nuclide Name	Half-Life	Nuclide Type	Key Line	Energy	Abundance
Ra-226	1602.00Y	natural		186.21 keV	3.28 %
				241.98 keV	7.49 %
				295.21 keV	19.20 %
				351.92 keV	37.20 %
				609.31 keV	46.30 %
Ra-228	6.70Y	natural		338.40 keV	12.01 %
				911.10 keV	29.00 %
				968.90 keV	17.50 %
Th-228	1.89Y	natural		238.60 keV	43.10 %
				583.20 keV	30.90 %
Th-234	1 000.00Y	natural		63.30 keV	3.81 %
				92.60 keV	5.42 %
U-235	7.04E+08Y	natural		143.76 keV	10.50 %
				163.35 keV	4.70 %
				185.71 keV	54.00 %
				205.31 keV	4.70 %
Np-239	2.35D	fission		106.13 keV	22.70 %
				228.19 keV	10.70 %
				277.62 keV	14.10 %
AM-241	432.20Y	activation		59.54 keV	35.70 %

Table 2. Calculation of Gamma Efficiency

CALIBRATED 01 NOV-93
5.1595 grams/sample

Nuclide	(keV)	Abund	gam/sec•g	$T_{1/2}$ Days	Spike gam/sec	cps[a]	EFF cps/gps[b]
Am-241	60	0.3570	1140	1.58E+05	5882	35.31	0.600
Cd-109	88	0.0370	652	4.64E+02	3324	35.35	1.064
Co-57	122	0.8551	597	2.71 E+02	3018	38.21	1.266
Ce-139	166	0.8035	690	1.38E+02	3420	41.23	1.206
Hg-203	279	0.8155	2023	4.66E+01	9267	81.23	0.876
Sn-113	392	0.0193	2115	1.15e+02	10399	70.65	0.679
Sr-85	514	0.9830	4021	6.48E+01	19046	105.64	0.555
Cs-137	662	0.8512	2499	1.10e+04	12887	59.88	0.465
Y-88	898	0.9340	6279	1.07E+02	30756	112.54	0.366
Co-60	1173	1.0000	3522	1.92e+03	18119	54.33	0.300
Co-60	1333	1.0000	3525	1.92E+03	18135	48.15	0.266
Y-88	1836	0.9938	6654	1.07E+02	32592	69.02	0.212

SPIKE = (gammas/sec • gram) • (@EXP(−O.693 • (T(count)−T(calib))/T(1/2))) • (grams/sample)
EFF = 100 • cps/SPIKE

[a] counts/sec
[b] gammas/sec

GROSS ALPHA AND BETA IN AIR

Analyte: Gross Alpha and Beta

Minimum Detectable Activity:*
80×10^{-5} pCi/m^3 for Alpha
240×10^{-5} pCi/m^3 for Beta

Procedure: Count the air filters with a gas proportional counter.

Matrix: Air

Analysis time: Approximately 24 samples per day

Accuracy: Should be within ± 20% of known value.

* These activities are based on 8 pCi of alpha and 24 pCi of beta for 10,000 cubic meters of air

Scope and Application

This procedure is for the preparation of 5- or 10-cm (2- or 4-inch) glass fiber air filters for alpha/beta gas-proportional counting.

Sample handling and preparation is applicable to 5 cm glass fiber air filters collected for the monitoring of environmental gross alpha and beta activities.

Summary of Method

This method provides an approximate measure of the alpha and beta activity of air filters. It is intended as a screening method and at best provides only approximate results. The method requires that the approximate detection efficiency of the instrument be known for both alpha and beta activity in air filters. Because the filters must not be covered during counting, special care must be taken to avoid cross contamination.

Sample Handling and Preservation

Store air filters in a plastic or glassine envelope until ready for counting.

Interferences

At collection, natural radioactivity will be present in substantial amounts for several days after sample collection. When counting samples within one week of collection, care should be taken to account for radon/thoron progeny.

Supplies

Disposable shallow dish planchets
Envelopes for air filters
Gas flow proportional counter
Marking penTweezers

1 Mark a counting planchet with the sample number. Carefully transfer the air filter "collected side up" to the numbered planchet using tweezers.

2 Place the labeled planchets into the proportional counter sample holder.

3 Count the sample for a period determined by the Laboratory Supervisor.

4 Calculated results may be obtained with computer software. Efficiency factors for both alpha and beta are required.

Calculations

Gross alpha activity is calculated as follows:

$$Activity\ Concentration\ \left(pCi/m^3\right) = \frac{C_s - C_b}{(2.22)(Eff)(Vol)}$$

where

Cs = sample counts/min.

C_b = background counts/min.

Eff = calibration efficiency

Vol = sample volume(m^3)

$$\sigma = (2)\ (Activity)\ \sqrt{\left[\frac{\frac{C_s}{T^2} + \frac{C_b}{T^2}}{\left(\frac{C_s}{T} + \frac{C_b}{T}\right)}\right]}$$

where

2σ = error (2 times sigma)

C_s = sample counts (gross)

C_b = background counts

T = counting time

$$MDC = \frac{2.71 + (4.65\sqrt{C_b}}{(2.22)(Eff)(Vol)(T)}$$

where:

MDC = Minimum Detectable Activity

C_b = background counts

T = counting time (must be same for sample and background)

Eff = calibration efficiency

Vol = sample volume (m^3)

Gross beta activity is calculated as follows:

- **If there is no significant alpha count** when the sample is counted at the alpha voltage plateau, the beta activity can be determined as follows:

$$Activity\ Concentration\ \left(pCi/m^3\right) = \frac{C_s - C_b}{(2.22)(Eff)(Vol)}$$

where

C_s = sample count/min

C_b = background count/min

Eff = beta efficiency factor

Vol = sample volume (m^3)

- **When counting beta radioactivity** in the presence of alpha radioactivity by gas-flow proportional counting systems (at the beta plateau), alpha particles are also counted. When significant alpha activity is indicated at the alpha plateau voltage, the beta activity of the sample can be determined by counting the sample at the beta voltage plateau and calculating the activity from the following equation:

$$Activity\ Concentration\ \left(pCi/m^3\right) = \frac{C_s - AE}{(2.22)(Eff)(Vol)}$$

where

C_s = net beta count rate (gross count rate minus the background count rate at the beta voltage plateau)

A = net alpha count rate (gross alpha count rate minus the background count rate) at the alpha voltage plateau.

E = alpha amplification factor, read from the graph of the ratio of alpha counted at the beta voltage/alpha counted at the alpha voltage

Eff = beta efficiency factor

Vol = sample volume (m^3)

The counting uncertainty and the MDA calculations are the same as those used for the gross alpha activity.

TRITIUM SCREENING METHOD FOR WATER

Analyte: Tritium

Minimum Detectable Activity:* 300–500 pCi/L, depending on counting time

Procedure: Add the raw water to liquid scintillation cocktail and count.

Matrix: Water

Accuracy: Variable, used as a screening method only, not for exact results.

Scope and Application

Caution

This is a screening method for tritium in water. It will also detect other alpha and beta emitters in water and therefore has some general application beyond tritium. If activity is detected, it may or may not be tritium. Therefore, results should be interpreted cautiously. This method is best used to rule out further analytical work if activity is not found.

- Do not use a water sample that is discolored.

If activity is found by this method, use the distillation method, "Tritium in Water by Distillation," to confirm the presence of tritium. If activity is found by this method but not by the distillation method, some other natural or artificial radionuclide is likely present.

The results and the behavior of the liquid scintillation counter can be routinely monitored by counting a tritium standard and background samples in the same batch as the prepared water sample.

Summary of Method

An aliquot of the raw water sample is taken and mixed with a liquid scintillation cocktail in the proportions recommended by the manufacturer of the cocktail. With the more recent nonhazardous cocktails, this usually requires 4–5 mL of sample and 16–15 mL of cocktail. Sample + Cocktail = 20 mL.

The scintillator solution, mixed with the radioactive sample, is excited by alpha/beta particles and emits light pulses by a molecular de-excitation process. The number of pulses per unit time is proportional to the quantity of activity present. Multiple solutes are used in the scintillator to provide the best combination of wavelength and pulse height for this application.

The pulses are detected by two photomultiplier tubes connected in coincidence and converted to electric signals. The amplified pulses are recorded and the count rate is measured. The efficiency of the system can be determined by use of prepared tritiated water standards having the same density and color as the sample.

Sample Handling and Preservation

Collect water sample in its natural state.

Do not acidify.

Since tritium in water is either in the form of T_2O or HTO, there is no need for special handling or preservation.

Interferences

Tritium in background water is an interference. Slightly elevated levels are present in surface waters, so deep well sources for background water should be used.

All fluors should be checked for excitation under lighting conditions being used and, if necessary, they should be exposed only to red light. Toluene- or xylene-based solutions are not used due to waste issues. Reagents are commercial, nonhazardous liquids scintillation solutions. Scintillators exposed to fluorescent lighting should be dark-adapted for a minimum of 1 hour.

The use of plastic vials may cause buildup of static charge and give erratic results. Tritium can also migrate over a period of time.

Apparatus

Coincidence-type liquid scintillation spectrometer

Liquid scintillation vials: Low-potassium glass is recommended. Polyethylene vials may be used when dioxane liquid scintillator solution is used.

Reagents

Background water with tritium activity below the minimum detectable activity (most deep well waters are low in tritium content).

Commercial, nonhazardous liquid scintillation solution

Procedure

These preparations vary in their sample carrying capacity. Normally, commercial solutions will take from 4 to 10 mL of water sample when the total volume of cocktail and sample is 20 mL. Use the proportions recommended by the manufacturer of the cocktail, and mix sample and cocktail.

1 Prepare background and standard tritium water solutions for counting, using the same amount of water and the same scintillator as used in the preparation of samples.

   Use low tritium background distilled water for these preparations (distillate of most deep well water sources is acceptable, but each source should be checked for tritium activity before using).

2 Dark-adapt all samples, backgrounds, and standards.

3 Count the samples, backgrounds, and standards at least long enough to meet the required detection limit for the sample.

Tritium is normally counted with a window setting where the figure of merit is at maximum.

Calculations

$$\text{Figure of Merit} = \frac{E^2 = (\text{Efficiency})^2}{B\ (\text{Background})}$$

Calculate the tritium concentration, A, in picocuries per liter as follows:

$$A = \frac{(C - B)\ (1000)}{(2.22\ )(E)\ (V)\ (F)}$$

where

C = sample count rate, cpm

B = background count rate, cpm

E = counting efficiency, as determined below

V = volume of the sample aliquot in ml

F = recovery correction factor, as determined

2.22 =conversion factor for dpm/pCi

Determine the counting efficiency, E, as follows:

$$E = \frac{D - B}{G}$$

where

D = distilled water standard count rate, cpm

B = background count rate, cpm

G = activity of distilled water standard (dpm)

Calculate the recovery correction factor, F, as follows:

$$F = \frac{L - B}{(E)\ (M)}$$

where

L = raw water standard distillate count rate, cpm

B = background count rate, cpm

E = counting efficiency, as determined in above

M = activity of raw water standard (before distillation), dpm

Calibrations

Determination of Recovery and Counting Efficiency Factors (See Calculations Section)

1 **Raw Water Tritium Standard Solution.** Into a 1 L volumetric flask, pipette a tritium standard solution containing approximately 1000 disintegrations per minute (dpm) per mL and dilute to volume using low level tritium background raw water (undistilled) and standard tritium activity. Label this solution "Raw Water Tritium Standard Solution."

For tritium background determinations, distill approximately 600 mL of water, obtained from the same raw water source as above (without tritium activity added). Using the distillate and standard tritium activity, prepare a tritium standard solution in a 500 mL volumetric flask containing the same specific activity as the "Raw Water Tritium Standard Solution." Label this solution "Distilled Water Tritium Standard Solution."

2 **For liquid scintillation counting**, prepare three aliquots of the "Raw Water Tritium Standard Solution."

- Distill three aliquots of the "Distilled Water Tritium Standard Solution" and three aliquots of the distilled raw water (for background).
- Mix appropriate distillate with liquid scintillator to give 20 mL total volume in a liquid scintillation vial.

  Some of the newer, nonhazardous commercial cocktails may require smaller volumes of sample. Follow the instructions of the manufacturer in those cases.
- Shake well and dark-adapt the vials overnight.
- Count each vial in a liquid scintillation counter long enough to meet the required detection limit.

Precision and Accuracy

This method is intended for screening only and therefore no attempt was made to determine precision and accuracy. Under ideal conditions, where the water is very clean and no other radionuclides are present, the accuracy should approach that of the conventional distillation method.

TRITIUM IN WATER BY DISTILLATION

Analyte: Tritium	**Matrix:** Water
Minimum Detectable Activity:* 300–500 pCi/L, depending on counting time	**Analysis time for 12 samples:** 2 days, including counting time
Procedure: Distill the sample and count	**Accuracy:** Good within ± 10% of known value.

Scope and Application

This method covers the measurement of tritium in a sample of drinking water by liquid scintillation counting. Although the method was intended for measurement of tritium in drinking water, the method is essentially the same as most methods for measurement of tritium in any water sample.

The results and the behavior of the liquid scintillation counter can be routinely monitored by counting a tritium standard and background samples at the same time as the prepared water sample.

Summary of Method

A 100 mL aliquot of a water sample is treated with a small amount of sodium hydroxide and potassium permanganate, distilled, and a specified fraction of the distillate is collected for tritium analysis. The alkaline treatment prevents other radionuclides such as radioiodine and radiocarbon from distilling over with the tritium. Some water will contain trace quantities of organic compounds (especially surface water sources that contain fish and other life). The permanganate treatment oxidizes trace organics in the sample aliquot which could distill over and cause quenching interferences.

A middle fraction of the distillate is collected for tritium analysis because the early and late fractions are more apt to contain interfering materials for the liquid scintillation counting process.

The collected distillate fraction is thoroughly mixed. A portion is mixed with liquid scintillator solution and, after dark adapting, is counted in the liquid scintillation counting system for tritium beta particle activity.

Sample Handling and Preservation

Note

The water sample should be collected in its natural state.

Do not acidify.

Since tritium in water is very likely to be in the form of T_2O or HTO, there is no need for special handling or preservation.

Interferences

Tritium in background water is an interference. Slightly elevated levels are present in surface waters, so deep well sources for background water should be used.

All fluors should be checked for excitation under lighting conditions being used, and if necessary they should be exposed only to red light. Toluene- or xylene-based solutions are not used due to waste issues. Reagents are commercial, nonhazardous liquid scintillation solutions. Scintillators exposed to fluorescent lighting should be dark-adapted for a minimum of 1 hour.

The use of plastic vials may cause buildup of static charge and give erratic results.

Apparatus

Coincidence-type liquid scintillation counter with alpha and beta spectrometry capabilities.

Liquid scintillation vials: Low-potassium glass is recommended. Polyethylene vials may be used when dioxane liquid scintillator solution is used.

Distillation apparatus: For aqueous distillation: 250 mL and 1000 mL round bottom pyrex flasks, connecting side arm adapter (such as Corning part #9060), condenser, graduated cylinder, boiling chips, and heating mantle.

Reagents

Reagents for distillation treatment: sodium hydroxide pellets and potassium permanganate (ACS – reagent grade).

Background water with tritium activity below the minimum detectable activity (most deep well waters are low in tritium content).

Commercially available scintillator solutions, preferably biodegradable. Refer to "Tritium Screening Method for Water," Interferences.

Procedure

1 Add 0.5 g sodium hydroxide and 0.l g potassium permanganate to a 100 mL aliquot of the sample in a 250 mL distillation flask.

- Add a boiling chip to the flask.
- Connect a side arm adapter and a condenser to the outlet of the flask, and insert a graduated cylinder at the outlet of the condenser.
- Heat the sample to boiling to distill, and discard the first 10 mL of distillate as a separate fraction.

It is important that the first 10 mL fraction for samples and standards alike be discarded, since there is a gradient in the tritium concentration of the distillate.

- Collect the next 50 mL of distillate for tritium analysis and mix thoroughly.

2 Mix appropriate distillate with liquid scintillator to give 20 mL total volume in a liquid scintillation vial.

3 Prepare background and standard tritium water solutions for counting, using the same amount of water and the same scintillator as used in the preparation of samples.

Use low tritium background distilled water for these preparations (distillate of most deep well water sources is acceptable, but each source should be checked for tritium activity before using).

4 Dark-adapt all samples, backgrounds, and standards.

5 Count the samples, backgrounds, and standards at least long enough to meet the detection limit required by the Laboratory Supervisor.

Note

Tritium is normally counted with a window setting where the figure of merit is at maximum.

Calculations

$$Figure\ of\ Merit = \frac{E^2}{B} = \frac{(Efficiency)^2}{(Background)}$$

Calculate the tritium concentration, A, in picocuries per liter as follows:

$$A = \frac{(C - B)\ (1000)}{(2.22)\ (E)\ (V)\ (F)}$$

where

C = sample count rate, cpm,

B = background count rate, cpm,

E = counting efficiency,

V = volume of the sample aliquot in mL,

F = recovery factor,

2.22 = conversion factor for dpm/pCi.

Determine the counting efficiency, E, as follows:

$$E = \frac{D - B}{G}$$

where

D = distilled water standard count rate, cpm,

B = background count rate, cpm, and

G = activity of distilled water standard (dpm)

Calculate the recovery correction factor, F, as follows:

$$F = \frac{L - B}{(E)\ (M)}$$

where

L = raw water standard distillate count rate, cpm,

B = background count rate, cpm,

E = counting efficiency, and

M = activity of raw water standard (before distillation), dpm.

Calibrations

Determination of Recovery and Counting Efficiency Factors (See calculations)

- **Raw Water Tritium Standard Solution.** Into a 1 L volumetric flask, pipette a tritium standard solution containing approximately 1000 disintegrations per minute (dpm) per mL and dilute to volume using low level tritium background raw water (undistilled) and standard tritium activity. Label this solution "Raw Water Tritium Standard Solution."

  For tritium background determinations, distill approximately 600 mL of water, obtained from the same raw water source as above (without tritium activity added). Using the distillate and standard tritium activity, prepare a tritium standard solution in a 500 mL volumetric flask containing the same specific activity as the "Raw Water Tritium Standard Solution." Label this solution "Distilled Water Tritium Standard Solution."

- **Aqueous permanganate distillation:** To a 100 mL aliquot of the "Raw Water Tritium Standard Solution" in a 250 mL distillation flask, add 0.5 g sodium hydroxide, 0.l g potassium permanganate, and a boiling chip.

  Continue according to the procedure described in Step 1 of Procedure section. Discard the first 10 mL and collect 50 mL of the distillate for analysis. Mix well. Repeat the distillation with two more 100 mL aliquots for triplicate analyses.

- **For liquid scintillation counting:**

  1 Prepare three aliquots of the "Raw Water Tritium Standard Solution" distillate, three aliquots of the "Distilled Water Tritium Standard Solution," and three aliquots of the distilled raw water (for background).

  2 Mix appropriate distillate with liquid scintillator to give 20 mL total volume in a liquid scintillation vial.

     Some of the newer, nonhazardous commercial cocktails may require smaller volumes of sample. Follow the instructions of the manufacturer in those cases.

  3 Shake well and dark-adapt the vials overnight.

Precision and Accuracy

This method is capable of providing data precise and accurate enough to meet the data quality objectives specified by the Laboratory Supervisor in either early or late stages of the release.

BETA SCREENING OF SMEARS

Sample Types: Wipes or smear samples including nasal smears **Geometry:** Liquid scintillation vial **Procedure:** Compare count results from liquid scintillation of samples and spiked samples	**Analysis time:** Approximately 1 hour including preparation and counting **Matrix:** Q-Tip or filter in liquid scintillation vial ***Accuracy:*** Good within ± 10% of known value

Scope and Application

This procedure is applicable to analysis of either filter or "Q-Tip" wipe tests.

The resulting data will screen for the presence of deposited alpha and beta emitters on surfaces or those deposited in nasal passages of personnel exposed to airborne radionuclides.

Summary of Method

Samples are placed into a scintillation vial with 20 mL of suitable scintillation cocktail that does not require dark adaption. The vial is capped, shaken, and counted. Counts from samples are compared to those of spiked samples counted in the same batch.

Supplies

Cellulose nitrate filter papers, 2"
Liquid scintillation vials, 20 mL with poly-seal caps
Liquid scintillator solution
Q-Tips
Radioactive standard solution appropriate to isotope(s) of interest

Procedure

Prepare background sample

1 Place a clean cellulose nitrate filter paper or a Q-Tip into an empty scintillation vial.

2 Add 20 mL of Packard Filter Count (or equivalent) scintillation cocktail. Cap the vial tightly and shake it for 20–30 seconds. Wipe off any fingerprints.

3 Place this vial in the first position in the sample batch.

Prepare Standard Performance Reference Material (SPRM)

1 Prepare two (2) SPRM samples for every ten samples or less.

2 Place a clean cellulose nitrate filter paper or Q-Tip into each of the duplicate scintillation vials.

3 Add 90–100 pCi of traceable radioactive standard (of the isotope of interest) to each vial.

4 Bring the volume to 20 mL by adding appropriate scintillation cocktail. Cap the vials tightly, shake them for 20–30 seconds, and wipe off any fingerprints.

5 Place one SPRM vial in the second position in the sample tray and the other vial immediately after the last sample to be analyzed.

Sample Preparation

1 Place the cellulose nitrate filter paper or the Q-Tip into a clean scintillation vial.

2 Add 20 mL of scintillation cocktail.

3 Cap the vial tightly, shake it for 20–30 seconds, and wipe off any fingerprints.

4 Place the sample vial(s) starting in the third position after the first SPRM. Place the second SPRM sample immediately after the last sample vial.

Sample Counting

1 Place the samples (background, SPRMs, and unknown samples) into the appropriate trays or sample locations in the liquid scintillation analyzer.

2 Count each vial for a time sufficient to meet data quality objectives.

Calculations

Calculate the activity of the sample in picocuries as follows:

$$Activity = \frac{C - B}{(2.22)\ (Eff)}$$

where

C = sample count rate, cpm

B = background count rate, cpm

Eff = counting efficiency

2.22 = conversion factor for dpm/pCi

Determine the counting efficiency, Eff, as follows:

$$Eff = \frac{D - B}{G}$$

where

D = standard in distilled water count rate, cpm

B = background count rate, cpm

G = activity of standard in distilled water, dpm

RADIOACTIVE KRYPTON AND XENON IN AIR BY CRYOGENIC SEPARATION

Analyte: Kr-85 and Xe-133

Minimum Detectable Activity:
5 pCi/m^3 at time of count for 0.5 m^3 sample

Procedure: Purify and isolate Krypton and Xenon by gas chromatography and count by liquid scintillation

Matrix: Air

Analysis time: 2 hrs.

Accuracy: Fair, ± 20%

Scope and Application

This is a procedure for the analysis of environmental air samples for Radiokrypton and Radioxenon and applies only to the apparatus shown in Figure 1.

Note

This is a specialized procedure and requires special handling. This procedure applies to all air samples.

Summary of Method

The stable noble gases krypton and xenon, present at the parts per million (ppm) level in the air sample, are purified and separated in the analytical process. Any radioactive krypton or xenon is carried with the stable isotopes and can be extracted with the stable elements. This analytical procedure is based on gas chromatography.

The krypton and xenon fractions are counted separately by liquid scintillation counting. The optimum sample size is approximately 0.5 cubic meters of air at standard temperature and pressure (STP). Smaller samples have less krypton, making the analysis more difficult and with greater statistical uncertainty. The minimum sample size is approximately 0.3 cubic meters.

Sample Handling and Preservation

Air samples are collected in pressurized containers. Except for normal safety precautions associated with compressed air, no special handling or preservation is required. The short half-life of radioactive xenon makes it necessary to analyze the samples within a few days of collection.

Interferences
Radon can sometimes be mistaken for Xe-133.

Apparatus and Supplies

Figure 1 shows a schematic of the noble gas analysis apparatus.

Apparatus

Bottles (cylinders) for sample collection
Charcoal (for sample collection traps)
Computer (for calculation, optional but preferred)
Copper tubing
Data cards, sample control supplies
Dewars (assorted sizes)
Dry ice maker
Fume hood
Gas valves (for vials)
Gas Chromotgraph (informally called "gas rig")
Glass vials (20 mL capacity) (custom made)
Glass traps-large and small molecular sieve traps (custom made)
Heat gun
Heat unit for traps
Heating mantle (for heating cocktail)
Immersion type heater
Ladle (for handling dry-ice acetone bath)
Pinch clamps
Pressure gauge for tanks (cylinders) (if not equipped with gauge)
Refrigerator
Scintillation counter
Syringes (50 & 30 cc)
Thermal gloves
Thermometers
Vacuum pumps
Water source (used in cocktail heating set up)

Supplies

Acetone
Dimethyl POPOP

Drierite (moisture absorption from sample)
Dry ice
Ethyl alcohol
Helium supply (this is carrier gas)
Liquid nitrogen
Molecular sieve 30/60 mesh (sample collection)
PPO
Toluene (for cocktail)

Preparation of Dry Ice-Acetone Bath

1 Fill large dewar approximately 1/2 to 3/4 full of acetone.

2 Using dry ice maker, make a block of dry ice.

3 Slowly add the dry ice to the dewar of acetone in very small pieces until acetone starts to cool down.

4 Stir liquid with ladle after the addition of each block of dry ice.

5 Continue to add dry ice until a slush forms. After the weekend it usually takes 3-4 blocks of dry ice. During the rest of the week it will be necessary to add 1-2 blocks of dry ice to maintain the slush.

6 Once slush forms, cover the dewar as a precaution against fumes.

7 Observe the following precautions:

- Wear insulated gloves and goggles when handling dry ice.
- Avoid exposure to acetone fumes.
- No open flames.
- Keep flask covered with sponge when not in use.

Preparation of Counting Cocktail

Note

It is important that the heating mantle for the cocktail be on and the cocktail hot before injecting it into the sample vial.

1 In a 4 L Erlenmeyer flask weigh out:

- 1.5 grams Dimethyl POPOP

- 6.0 grams PPO

2 Add 4 L of scintillation grade toluene.

3 Stir until Dimethyl POPOP and PPO are completely dissolved.

4 Pour prepared cocktail into 1 L round bottom flask to heat it for use in the sample collection procedure.

Calibrations

The sample counting efficiency is determined by counting a vial containing a known amount of Krypton-85. The computer calculations correct for the decay of the Kr-85 standard.

Procedure

1 **Lab personnel recheck the pressure of each sample bottle** before analysis, even though it was recorded in the field.

If the pressure is more than 10% lower than listed on the card (tag), check valve for leaks by immersing it in a pan of water. Install a new valve if necessary. If a sample has been lost in the field, enter "insufficient sample" in data base. For samples lost in the lab, etc., enter "sample lost" in data base.

2 **Weigh the full sample bottle** and record the weight on the data card.

If the bottle pressure is greater than 200 psi for the bottles, a single bottle will provide enough sample for the analysis. If the pressure is less than 200 psi, both bottles must be used. Weigh the empty container and record the weight on the data card. The weight of the sample is the difference between the two weights.

3 **Prepare the gas rig** to receive a new sample by baking out the charcoal trap at 350°C

The large charcoal trap is normally in a prepared condition since previous samples have been removed by baking. If the valves to this trap have been closed, then no preparation is necessary for this trap. If the valve was left open, air can first be removed by using the vacuum and then baking the trap while a helium flow is maintained. It is important not to bake the charcoal trap in the presence of room air (i.e., with outside vent open), as this will ruin the charcoal.

The last (molecular sieve) trap must be baked out on the rig while a helium flow is maintained. After 45–60 minutes, remove the heating furnace and close the helium vent valve. All vents on traps should be closed following bakeout to keep out room air. Place a cylinder of drierite in the inlet line to remove moisture from the sample. Replace this material when the blue indicator color fades.

4 **Loading sample onto charcoal.** Immerse the large charcoal trap in liquid nitrogen just before loading the sample. Attach sample to rig at appropriate site. Open valves 5 and 7. Open valve on sample bottle and draw sample onto charcoal by means of the pressure difference. Watch the pressure reading; it should not exceed 500 mm of mercury. If pressure is too great, it could cause liquid air to form and create an explosive mixture if liquid air reaches the charcoal.

If you need to leave the room during this phase, stop loading the sample and close all valves leading into and out of the large charcoal trap. If, while loading the sample, the pressure should exceed 500 mm of mercury for more than a few seconds, check the precooler for liquid air.

5 **Degassing.** Once the sample is loaded onto the charcoal, close valves 5 and 7. A gross type of separation, referred to as degassing, is the separation of the majority of oxygen and nitrogen from the sample at −80°C.

Replace the liquid nitrogen bath around the charcoal trap with a dry ice-acetone bath, while maintaining a helium flow (by opening valves 9, 10, and 11). The degassing process takes 1–1.5 hours. Helium is being vented during this step.

6 **Heating.** 15 to 20 minutes prior to initiating the heating phase, slowly raise the liquid nitrogen container around the convoluted trap containing the molecular sieve. Close valve 11 and open valves 13 and 15. (Do not close values 9 and 10.)

The charcoal trap is heated to drive the sample to the trap containing the molecular sieve by way of the helium flow. The heating phase takes 20 minutes and ensures that the entire sample is transferred.

7 **Krypton-85 is separated from the remaining impurities by elution at a temperature of about −35°C.** This phase separates the noble gases from the partially purified sample. The sample is now in the molecular sieve trap.

First, close valves 9, 10, 13, and 15. While the sample is submerged in liquid nitrogen, open valves 14 and 15. Check the helium flow meter to ensure adequate flow rate.

Replace the liquid nitrogen with an ethanol bath at a temperature of −35°C to −30°C. The alcohol bath is prepared by cooling a small dewar (approximately 1200–1600 mL capacity) with liquid nitrogen.

Once the heater is in place, pour the liquid nitrogen out of the dewar and fill the dewar 2/3 full of ethyl alcohol which has been stored in a freezer. The alcohol is stored in the freezer to help lower its temperature to –35°C more quickly. Check the temperature of the alcohol before using it. The molecular sieve trap, having just emerged from liquid nitrogen, will lower the alcohol temperature to about –45°C. Turn on the chart recorder.

8 **Elution.** Submerge the copper loop located to the right of the molecular sieve trap in liquid nitrogen. Maintain liquid nitrogen levels. Attach the small glass sample vial to the appropriate connection site located to the right of the copper loop.

Note

You can open the charcoal trap to vacuum, valves 9, 10, and 11, with the heating furnace operating for an additional 20 minutes. This ensures that the charcoal trap is purged of the sample and ready for re-use.

Oxygen should elute quickly, as indicated by the peak on the chart recorder. After the oxygen peak has passed and the pen on the recorder has returned to the base line, switch the helium flow through the copper loop to collect the krypton fraction.

9 **Second Helium Venting.** After the krypton has passed as indicated by the chart recorder, switch the helium flow to vent once again. This will open the copper loop to the vacuum process which removes traces of helium. Close the vacuum valve and remove the liquid nitrogen bath from the wire loop. Using the heat gun, drive the krypton from the copper loop to the small glass vial that has been previously evacuated. Record the pressure reading from the manometer.

10 **Xenon collection.** Attach the second vial to collect xenon. Replace the liquid nitrogen around the copper loop. Replace the ethanol bath water with a water bath containing a wand heating element. Soon after the krypton peak has passed (3–5 minutes), the nitrogen peak can be seen on the recorder; allow this to vent out.

Switch the helium flow through the copper loop to collect the xenon fraction. Unplug the water bath when the water reaches the boiling point. After the xenon peak has passed, as indicated by the recorder, once again switch the flow of helium back through the vent. Close the vacuum valve. Remove the liquid nitrogen from the copper loops and heat the loops with a heat gun to drive the sample to the collection vial. Close the valve on the vial and record the pressure reading.

Secure the rig and proceed to the hood to inject the scintillation cocktail into sample vials. Press the small plastic cap onto the sample vials. The samples are now ready to be placed on the counter.

11 **Place numbered vials in the corresponding sample holders** on the detector and record the placement number on the sample data card. Dark adaptation of the samples is not required for this procedure.

12 **Count each sample for 200 minutes** on a Beckman LS-3133T liquid scintillation counter or equivalent.

Rig Shutdown

1 Shut off all vacuum pumps.

2 Close all valves (helium supply and vent).

3 Release pressure on helium regulator.

4 Open any set of helium supply and vent valves to bleed helium from the line.

5 Remove the large trap of the molecular sieve from the liquid nitrogen and lay on the counter top.

6 Reclose all open valves.

7 Turn off helium in compound.

8 Check to see that plastic caps are on the vial attachment port.

9 Check to see that chart recorder and power supply are shut off.

Calculations

Krypton-85

$$V_{Kr}(mL) = \frac{(v)\ (p)\ (273)}{(760)\ (273 + t)}$$

where

V_{Kr} = volume of krypton in vial

v = vial volume (mL)

p = vial pressure (mm)

t = temperature (degrees Celsius)

$$Krypton\text{-}85\ \left(pCi/m^3\right) = \frac{A}{(2.22)\ (C.E.)\ \left(V_{Kr}\right)\ (S)\ (Y)}$$

where

A = gross cpm-bkg cpm

2.22 = $C. E.$

$C.E.$ = fractional counting efficiency

V_{kr} = volume krypton counted (mL)

$$S = 1293 \frac{\text{weight of sample}}{(\text{weight in grams of 1 m}^3 \text{ dry air})}$$

Y = 1/(l. 1 4) (S)

Xenon-133

$$V_{Xe}(mL) = \frac{(v)(p)\ (273)}{(760)\ (273 + t)}$$

where

V_{Xe} = volume of xenon in vial

v = volume of vial (mL)

p = vial pressure (mm)

t = temperature (degrees Celsius)

$$Xenon\text{-}133\ or\ Xenon\text{-}135(pCi/m^3) = \frac{(A)\ (Vc_{Xe})}{(2.22)\ (C.E.)\ (S)\ (V_{Xe})}$$

where

A = gross cpm-bkg cpm

Vc_{Xe} = volume Xe carrier added (mL)

2.22 = dpm/pCi

$C.E.$ = fractional counting efficiency

S = sample size (m3)

V_{xe} = volume xenon counted (mL)

Quantitative/ Qualitative Criteria

Results for krypton-85 and xenon-133 are normally constant in the atmosphere. Kr-85 is usually between 20 and 30 pCi/m^3, while xenon activity is normally not detectable. Spiked samples should be within 20% of the known value and blanks are below the detection limits, and are essentially zero.

Limitations

The optimum sample size is approximately 0.5 cubic meter of air. For samples of less than 0.3 cubic meters, the krypton peak may become too small to recognize.

Cleaning Noble Gas Sample Vials

Note: Use a fume hood.

1 Remove cocktail from vial using the syringes or vacuum pump.

Dispose of waste cocktail in a properly labeled container as specified in the Chemical Hygiene Plan or by the MD Manager.

2 Using a syringe, inject ethyl alcohol into vials for the first alcohol rinse and shake gently.

3 Remove alcohol from vials using syringe or vacuum pump.

4 Using syringe, inject soapy water or Isoclean into vials. Clean in the ultrasonic cleaner for 15 minutes.

5 Remove soapy water/Isoclean as with alcohol, above.

6 Using syringe, inject distilled or de-ionized water into vials to rinse. Repeat this step as many times as needed to ensure complete removal of all soap. Remove water as with alcohol, above.

7 Inject alcohol into vials for second and final alcohol rinse. Repeat steps 2 and 3. Be sure to remove as much of the alcohol as possible.

8 Place vials on metal tray and place in drying oven. The door of the oven should be left slightly ajar to vent for 20–30 minutes at the beginning of the drying cycle.

9 Dry vials for at least 1.5–2 hours at a temperature of at least 100°C.

Checking for radioactive buildup

Sample vials will be checked at least quarterly to ensure that no gradual buildup of radioactivity escapes the cleaning process.

- Check the vials by filling with cocktail and counting for 1 hour or more. If the count rate is more than 10% higher than the normal backgrounds taken at the same time, remove the vial from use. Before returning the vial to use, it must be cleaned and successfully retested by the same method.

- Segregate vials that are not suitable for use and tag them as defective to prevent accidental use. Records of testing will be maintained in a laboratory notebook or in other permanent records.

Loading Xenon Carrier Vials

- The petcock positions on the glass xenon handling apparatus are numbered and must be set at the following positions:

  [1] Closed
  [2] Open
  [3] Closed
  [4] Open
  [5] Open
  [6] Closed
  [7] Closed

- Turn on the vacuum pump. Check to be sure the valves on the xenon bottle are both closed. Turn on the vacuum gauge.

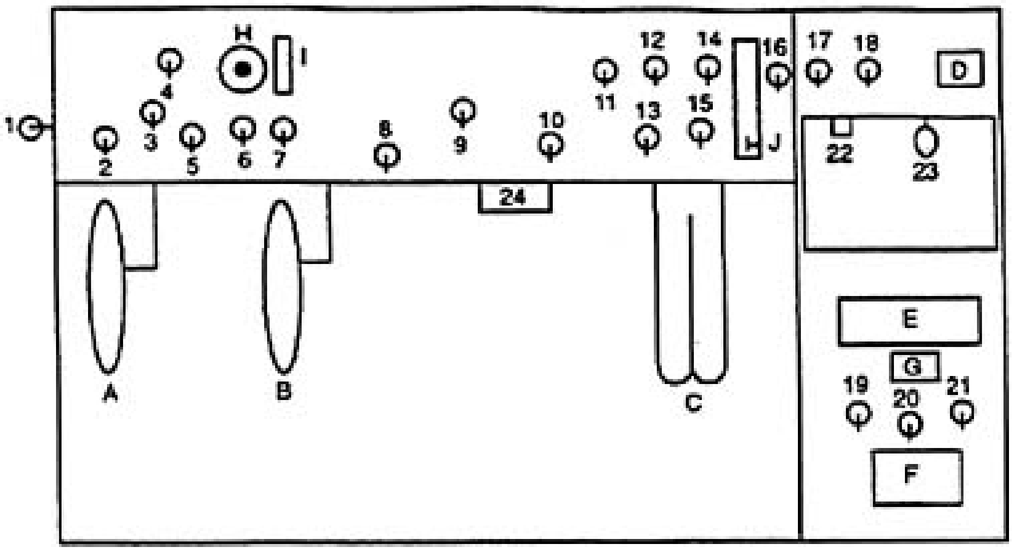
- Lightly grease 8 carder vials and set them in position with clamps. As vacuum increases, move vials slightly to ensure a good seal. Be sure arrows on vials face upward so that the vials are open to vacuum.

- Open Petcock 6 slowly, counterclockwise, red point down. Pull vacuum for approximately 15 minutes. The vacuum gauge should read 100 microns or less. Check that both mercury columns are at equal height.

- Close Petcock 5. Slowly open left valve at xenon bottle slowly. Allow mercury level to rise to 51 cm and close xenon valve.

- Close Petcock 6. Then close all xenon carrier vials with 1/4 turn.

- Open Petcock 5. Remove vials, wiping off any excess vacuum grease.



LEGEND:

- A Molecular sieve
- B Charcoal trap
- C MS–1
- D Manometer
- E Power supply
- F Chart recorder
- G Thermistor indicators
- H Vacuum indicator
- I Flowmeter
- J Flowmeter

NUMBERS:

- 1–21 Assorted valves
- 22 Wire loop (small charcoal trap)
- 23 Vial (sample collection site)
- 24 Degassing and heating site

FIGURE 1. NOBLE GAS ANALYSIS APPARATUS.

APPENDIX A
QUALITY ASSURANCE CERTIFICATION

Example 1: NIST-Traceable Standard Certificate File

CERTIFICATE OF CALIBRATION
GAMMA STANDARD SOURCE

Radionuclide:	Th-228	Customer:	BECHTEL NEVADA	
Half Life:	1.912 ± 0.002 years	P.O. No.:	VISA	
Catalog No.:	GF-228	Reference Date:	1 Jun 98	12:00 PST
Source No.:	556-90	Contained Radioactivity:	9.338	µCi
		Contained Radioactivity:	345.5	kBq

Source Description

a. Capsule type:	D
b. Nature of active deposit:	Incorporated into a ceramic matrix
c. Active diameter/volume:	5 mm
d. Backing:	Epoxy
e. Cover:	Acrylic

Radioimpurities:

None detected (other than daughters)

Method of Calibration

This source was assayed by gamma spectrometry:

Energy peak(s) integrated under:	277, 300, 583, 727.3, 860	keV.
Branching ratio(s) used:	0.0230, 0.0325, 0.3060, 0.0669, 0.0450	gamma rays per decay.

Uncertainty of Measurement

a. Systematic uncertainty in instrument calibration:	± 3.0%
b. Random uncertainty in assay:	± 0.7%
c. Random uncertainty in weighing(s):	± 0.0%
d. Total uncertainty at the 99% confidence level:	± 3.1%

NIST Traceability

This calibration is traceable to the National Institute of Standards and Technology.

Leak Test(s)

See reverse side for Leak Test(s) applied to this source.

Notes

1. IPL participates in an NIST measurement assurance program to establish and maintain implicit traceability for a number of nuclides, based on the blind assay (and later NIST certification) of Standard Reference Materials (As in NRC Regulatory Guide 4.15).

QUALITY CONTROL

22 May 98

Date Signed

ISOTOPE PRODUCTS LABORATORIES
1800 N. KEYSTONE STREET
BURBANK, CALIFORNIA 91504
818•843•7000 FAX 818•843•6168

Example 2: Radionuclide Decay Corrections FRMAC Gamma Nuclides

Today's Date: DD-MMM-YYYY					Time: 0000 (military time)		
Nuclide	Source	Reference Activity	Reference Date	Time	T 1/2 in Days	Corrected Activity	Total Activity in 5-mL Ampule
Co-57	2582-2	46.6 nCi/g	27-DEC-1991	1430	272	5.04 nCi/g	25.2 nCi
		SHADED	*SECTION*	*FOR*	*EXAMPLE*	*ONLY*	

Revised August 1998

Example 3: Preparation of Mixed Gamma Quality Assurance Samples

Certification	
NIST-Traceable Certification #:	**Supply copy of certificate to FRMAC Laboratory Supervisor**
Distribution:	
Reference Date: DD-MMM-YYYY **Time:** 0 0 0 0 (military time)	

Stock Solution (example of volume)	
Certificate #:	**Vol/Mass NIST Standard Used:**
Stock Solution #:	**Final Vol/Mass of Stock:**
Reference Date: DD-MMM-YYYY **Time:** 0 0 0 0 (military time)	**Balance #:**
Preparation Date: DD-MMM-YYYY **Time:** 0 0 0 0 (military time)	

Dilution from Stock Solution (example of volume)	
Preparation Date: DD-MMM-YYYY **Time:** 0 0 0 0 (military time)	**Vol/Mass Stock Solution Used:**
Stock Solution#:	**Final Volume:**
Dilution Solution#:	

Revised August 1998

APPENDIX B
EFFICIENCY PLOTS

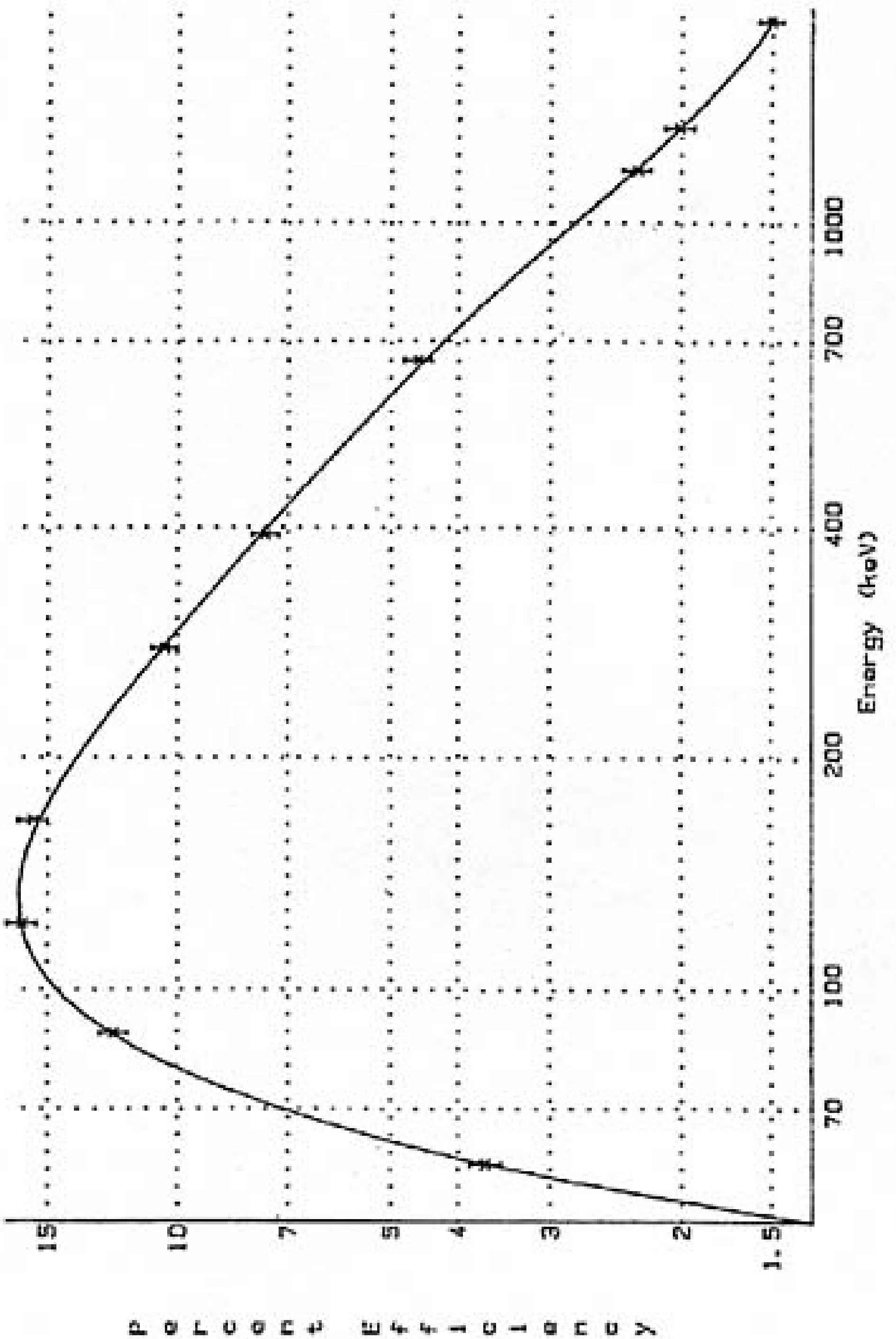


FIGURE B-1. ***HPGE COAX 26% WITH 2-INCH AIR FILTER***

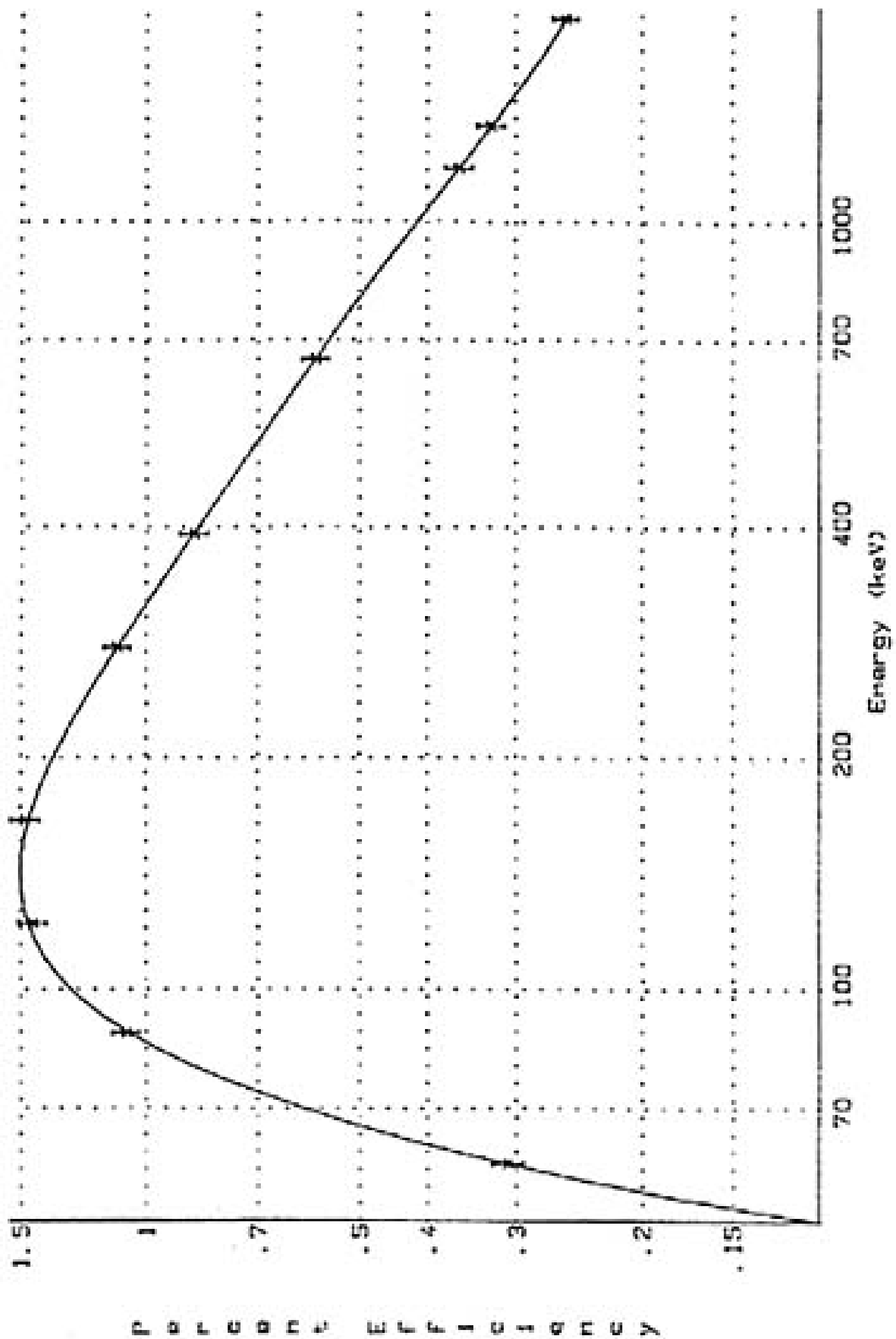


FIGURE B-2. ***HPGE COAX 28% WITH 3.5 L MARINELLI GEOMETRY***

APPENDIX C
FRMAC STANDARD UNITS

The primary reason for establishing Standard Reporting Units for FRMAC operations is to enhance communications and minimize the occasions for errors. The use of common units by all groups ensures all results presented to decision makers are consistent and understandable and that decision trees and tables are easily applied.

Radiological Units

Dose	rem	Sv
Exposure Rate:		
Closed window	mR/h	(mGy/h)
Open window	mR/h	(mGy/h)
Surface isotopic deposition	mCi/m^2	(MBq/m^2)
Air flow – detectors	cfm	(m^3/min)
Volume activity concentrations:		
Air Concentrations	pCi/m^3	(Bq/m^3)
Liquids	pCi/L	(Bq/L)
Soils	pCi/g	(Bq/g)
Vegetation	pCi/kg	(Bg/kg)
Tissue	pCi/kg	(Bg/kg)
Contamination:		
Surfaces	dpm/100 cm^2	(Bq/100 cm^2)
Swipe samples	dpm/100 cm^2	(Bq/100 cm^2)

Units of Measure

Metric units will be used as the FRMAC standard. At times, English units will be included for reference or additional information.

Time

Time will be reported as <u>local time</u> at FRMAC location in <u>military units</u> followed by the time zone. Example: 1415 EST (Eastern Standard Time).

Date

Dates will be reported as Day/Month/Year, MM-DDD-YYYY

Example: 07-Jul-1991

Meteorological

Wind speed:	miles per hour (mph)
Wind direction:	From in degrees (closest 5 degrees) (true North)
Wind elevation:	in feet above ground level (AGL) or above mean sea level (MSL)
Temperature:	degrees, °F

Map Orientation

Map and/or photo orientation will always be with TRUE North straight up.

Location

The location of all monitoring data will be reported in latitude and longitude units, with error terms, when available, AND physical description (street corner, road junctions, etc.) for map location.

APPENDIX D
LABORATORY CAPABILITY SUMMARY

LABORATORY CAPABILITY SURVEY

Page 1 of 7

Date:________________ Event:____________________

Laboratory Name/ID:		
Fixed ☐		Mobile ☐
POC:		
Phone:	Pager:	FAX:
Alternate POC:		
Phone:	Pager:	FAX:
Mailing Address:		
City:	State:	Zip Code:
Street Address:		
City:	State:	Zip Code:
MAXIMUM RADIOACTIVITY LAB WILL ACCEPT:		

Key Personnel

Name	Position	Phone

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LABORATORY CAPABILITY

Gross α/β Counting

Page 2 of 7

Date:________________ Event:____________________

Laboratory Name/ID:	
Instrument Manufacture:	
Model Number:	
Serial Number:	
Upgrades:	
Low background anti-coincidence counting?	Yes ☐ No ☐
Number of 2-in detectors:	
Count time for 2-in detector:	
Daily throughput for 2-in detector:	
Alpha (α) Filter Efficiency:	
Alpha (α) Filter Background:	
Beta (β) Filter Efficiency:	
Beta (β) Filter Background:	
Automatic sample chang[illegible]	Yes ☐ No ☐
Simultaneous alpha/beta count[illegible]	Yes ☐ No ☐
Instrument Manu[illegible]ure:	
Model [illegible]	
Se[illegible]Number:	
[illegible]rades:	
Low background anti-coincidence cou[illegible]g?	Yes ☐ No ☐
Numbe[illegible]ctors:	
Count time fo[illegible] detector:	
Daily throug[illegible]ut for 5[illegible] detector:	
[illegible]r Efficiency:	
[illegible]lpha ([illegible]ilter Background:	
[illegible]eta ([illegible] Filter Efficiency:	
[illegible]ta [illegible]Filter Background:	
[illegible]tic sample changer?	Yes ☐ No ☐
Simultaneo[illegible]s alpha/beta counting?	Yes ☐ No ☐

Calibration sources available? Yes ☐ No ☐

α source: Type: NIST Std: Yes ☐ No ☐ ID #: Activity:

β source: Type: NIST Std: Yes ☐ No ☐ ID #: Activity:

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LABORATORY CAPABILITY
Liquid Scintillation Counting

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Date:____________ Event:________________

Laboratory Name/ID:			
Instrument Manufacture:			
Model Number:			
Serial Number:			
Upgrades:			
Automatic sample changer?	Yes ☐ No ☐		
Background shielding?	Yes ☐ No ☐		
Quench correction?	Yes ☐ No ☐		
Visual energy spectrum?	Yes ☐ No ☐		
Number of programmable windows:			
Cocktail types available:			
Typical background/given time period:			
Estimated LLD with ^{3}H window completely open:			
Calibration sources available?	Yes ☐ No ☐		
Type:	NIST Std: Yes ☐ No ☐	ID #:	Activity:
Type:	NIST Std: Yes ☐ No ☐	ID #:	Activity
Type:	NIST Std: Yes ☐ No ☐	ID #:	Activity:
Type:	NIST Std: Yes ☐ No ☐	ID #:	Activity:

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LABORATORY CAPABILITY
Gamma Spectrometry

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Date:________________ Event:________________________

Laboratory Name/ID:

Detector Type	Manufacture	Model #	Serial #	Size	Efficiency	Shielding	Shielding Type/Size
1						Yes ☐ No ☐	
2						Yes ☐ No ☐	
3						Yes ☐ No ☐	
4						Yes ☐ No ☐	
5						Yes ☐ No ☐	
6						Yes ☐ No ☐	

MCA Manufacture	Model #	Serial #	Software	Upgrades
1				
2				
3				
4				
5				
6				

Calibrated Geometries

	Type	Geometry	Size/Volume	Container	Software
Filter					
Liquid					
Solid					
Gas					
Cartridges					

MCA inputs/# channels:

Data Reduction

Able to recalibrate for new [illegible] shield?	Yes ☐	No ☐
Listing of Gamma [illegible] available?	Yes ☐	No ☐
Able to change [illegible] libraries?	Yes ☐	No ☐

Sodium Iodide Screening

[illegible] screening?	Yes ☐	No ☐
Number of [illegible]detector/MCA units:		
Sizes:		
Shielding?	Yes ☐	No ☐
Shielding type/size:		
Screen Filters?	Yes ☐	No ☐
Screen Liquids?	Yes ☐	No ☐
Screen Solids?	Yes ☐	No ☐

Comments: ____

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LABORATORY CAPABILITY

Gamma Spec Cal Sources

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Date:________________ Event:__________________

Laboratory Name/ID:					
Gamma Spectrometry Calibration Sources					
	Type	Geometry	Nuclides in Source	Manufacturer	NIST Certificate Date
Filter	1				
	2				
	3				
	4				
Liquid	1				
	2				
	3				
	4				
Solid	1				
	2				
	3				
	4				
Gas	1				
	2				
	3				
	4				
Cartridge	1				
	2				
	3				
	4				

EXAMPLE

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LABORATORY CAPABILITY
Other Resources

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Date:________________ Event:__________________

SAMPLE

Laboratory Name/ID:				
Personnel				
Instrument Operators:	Yes ☐	No ☐	#Needed ________	
Systems analyst:	Yes ☐	No ☐	#Needed ________	
Data Reduction Technicians:	Yes ☐	No ☐	#Needed ________	
Sample Prep				
Standards Preparation Facility	Yes ☐	No ☐	#Needed ________	
If Yes, list types and sizes: ________________________				
Sample containers:	Yes ☐	No ☐	#Needed ________	
Swipe supplies:	Yes ☐	No ☐	#Needed ________	
Planchets:	Yes ☐	No ☐	#Needed ________	
Filters:	Yes ☐	No ☐	#Needed ________	
Other________________________:	Yes ☐	No ☐	#Needed ________	
Other________________________:	Yes ☐	No ☐	#Needed ________	
Other________________________:	Yes ☐	No ☐	#Needed ________	
Other________________________:	Yes ☐	No ☐	#Needed ________	
Other________________________:	Yes ☐	No ☐	#Needed ________	
Generator Power:	Yes ☐	No ☐	#Needed ________	
If Yes, number of units:__________ Capacity:__________				
Filtered hood exhausts:	Yes ☐	No ☐	#Needed ________	
Liquid nitrogen supply:	Yes ☐	No ☐	#Needed ________	
Reagent grade water:	Yes ☐	No ☐	#Needed ________	
[illegible]:	Yes ☐	No ☐	#Needed ________	
Computer Hardware/Software				
Desktop computer:	Yes ☐ No ☐	Brand:____________	Speed:________	#Needed ________
Laptop:	Yes ☐ No ☐	Brand:____________	Speed:________	#Needed ________
Printer:	Yes ☐ No ☐	Brand:____________	Speed:________	#Needed ________
Modem:	Yes ☐ No ☐	Brand:____________	Speed:________	#Needed ________
Network Card:	Yes ☐ No ☐	Brand:____________	Speed:________	#Needed ________
Spreadsheet:	Yes ☐ No ☐	Brand:____________		#Needed ________
Lab Database:	Yes ☐ No ☐	Brand:____________		#Needed ________
Statistics:	Yes ☐ No ☐	Brand:____________		#Needed ________
Word Processing:	Yes ☐ No ☐	Brand:____________		#Needed ________
Graphics:	Yes ☐ No ☐	Brand:____________		#Needed ________
Sample Management:	Yes ☐ No ☐	Brand:____________		#Needed ________
Sample Tracking:	Yes ☐ No ☐	Brand:____________		#Needed ________

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LABORATORY CAPABILITY
Other Resources Continued

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Date:________________ Event:__________________

Laboratory Name/ID:			
FAX Capabilities:	Yes ☐	No ☐	#Needed ________
If Yes, Number:			
Telephone (Land Line):	Yes ☐	No ☐	#Needed ________
If Yes, Number:			
Mobile Phone:	Yes ☐	No ☐	#Needed ________
If Yes, Number:			
Secure Phone:	Yes ☐	No ☐	#Needed ________
If Yes, Number:			
Pager:	Yes ☐	[illegible]	#Needed ________
If Yes, Access Number and/or Pin Number:			
Copy Machine:	[illegible] ☐	[illegible] ☐	#Needed ________

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